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NICKEL ALLOY STEEL FORGINGS

By Charles McKnight

Abstract

Large forgings constitute an important and constantly increasing field for alloy steel. The information regarding them is not nearly as complete or as readily available as the information pertaining to other forms of alloy steel. For example, the data published by the Society of Automotive Engineers on the properties, analyses and heat treatments of alloy steel are standard, but are not applicable to large forgings as they are based on tests made on one inch bars.

It is the purpose of this paper to deal briefly with the manufacture, uses, analyses, heat treatment and properties of nickel alloy steel forgings larger than four inches in diameter or equivalent section. It will not convey any helpful knowledge to the manufacturer of forgings, principally because most that it contains has been gleaned from them. To the prospective purchaser and user it may be helpful.

Uses

At the end of the last century when nickel steels first began to be used commercially the largest field of application was large forgings, such as armor, guns and shafting for naval vessels. Back in the 'nineties there were made for the Niagara Falls hydroelectric generators some weldless 3.0 per cent nickel steel field rings 12 feet in diameter and weighing 14 tons apiece—masterpieces then,

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but small compared to some modern forgings. Today, while ordnance consumes relatively few forgings, the industrial demand is steadily growing in pace with the increase in the size of engineering units. Forged alloy steel in now employed for large shafts, turbine motor and generator drums, gears, flywheels, pressure chambers, die blocks, Diesel engine parts, locomotive forgings, axles, extrusion dies, rolls and parts of steel mills, steam-hammer piston rods, hydraulic press rams, etc. In general, large forgings of this kind are used for parts of heavy machinery where strength and toughness, high impact and fatigue strength and reliability is required.

Manufacture

It is usually specified that the steel may be made by any process—acid or basic open-hearth, acid or basic electric furnace. Each process has some advantages and there is not an overwhelming argument advanced for any one. The electric furnace, however, does not contribute a large proportion, due to the comparatively small size of most electric furnaces. As for the addition of alloys to the steel, any reputable steel-maker with experience in this line is competent to make any of the alloy steels.

The questions of size and type of ingot mold, bottom or top pouring, use of hot-top, use of deoxidizers and cropping of the ingot are usually left to the manufacturer as he is responsible eventually for the quality of his product. It is general practice to proportion the ingot mold to the size of bloom or forging to be produced from it; forging molds are usually fluted; most molds are top-poured; a hot-top is used; a generous crop (20 per cent) is considered requisite and deoxidizers are sparingly used in the ladle and never in the mold.

The question of reduction from ingot to bloom and from bloom to forging has been a moot point for some time. As regards extremely large forgings, it seems almost to settle itself, as unquestionably it is preferable to be satisfied with a smaller ratio of reduction than to run the risks involved in the use of extraordinarily large ingots with the consequent liability to segregation, cooling cracks and dendritic structure. Further, the higher the ratio of reduction the greater the difference between the longitudinal and transverse properties of the steel.

Similarly, there always has been some controversy as to the

relative merits of hammering, pressing and rolling. The first two processes are used in the production of the actual forging; rolling is for the production of the bloom, intermediary between the ingot and the forging. Pressing is usually considered superior to hammering but, again, the quality of the end product is the real answer. Some hold that the rolled bloom is superior to the forged bloom. There is, however, little dispute on one point. Blooms of alloy steel destined to be reforged should be allowed to cool extremely slowly to avoid internal ruptures. It is quite general now to bury these blooms in ashes immediately after rolling or forging.

Effect of Alloys

The alloys used in forging steels are nickel, chromium, molybdenum and vanadium. Each, added by itself, confers certain properties on steel. By a combination of one or more, where each com-

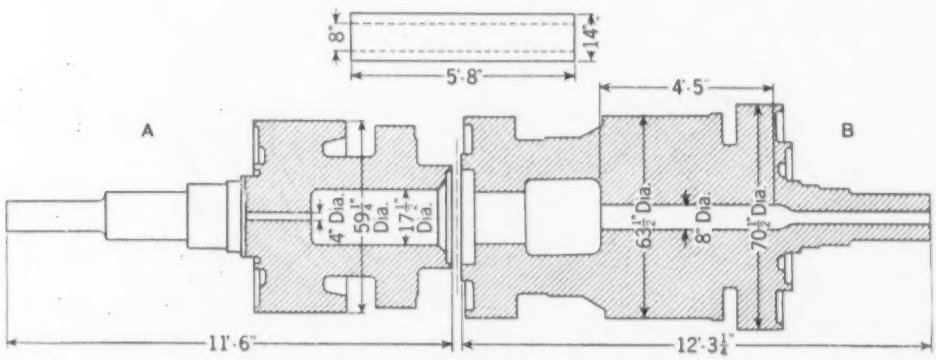


Fig. 1—Comparison of Dissimilar Heavy Forgings.

plements the other, it is possible to produce properties not obtainable by the addition of any one element alone—thus, as a prominent metallurgist recently observed, adding two and two and obtaining six as an answer.

Nickel is added to steel primarily to obtain increased strength with toughness. It is an element and a metal, similar to iron when added to steel it completely dissolves in the iron (ferrite) constituent and therefore its action is relatively independent of carbon as regards tensile strength, although the higher the carbon the greater the elastic ratio. This is shown in Table I.

Nickel is added to the steel bath in its metallic form, either as pig nickel or electrolytic sheets of almost chemically pure nickel.

Table 1

Carbon Content	Tensile Strength 3% Ni Steel	Tensile Strength Carbon Steel	Elastic Limit 3% Ni Steel	Elastic Limit Carbon Steel	Effect of 1% Nickel T. S.	Effect of 1% Nickel E. T.	Elastic Ratio Nickel Steel	Elastic Ratio Carbon Steel
0.20	85,000	55,000	48,000	28,000	+8571	+5714	56.5	50.9
0.30	95,000	75,000	60,000	37,000	+5714	6571	66.6	49.3
0.40	110,000	85,000	72,000	43,000	7142	8285	65.4	50.6
0.50	125,000	95,000	85,000	48,000	8571	10570	68.0	50.5

It does not oxidize, does not contaminate the bath, as it has no impurities, and is recoverable from the scrap. Particularly important in the manufacture of large forgings, it has recently been established that nickel has a tendency to render steel more homogenous and to lessen segregation. This is contrary to the prevalent belief.

Chromium is added primarily to harden and strengthen the steel.

NICKEL ALLOY FORGING STEELS

Type	Typical Analyses Carbon	Manganese	Nickel	Chromium	Molybdenum	Vanadium	Remarks
3% Nickel	0.13-0.50	0.40-0.80	Min. 3.00	..	..	..	General structural for strength and toughness.
2.5% Nickel	0.18-0.35	0.60-0.90	Min. 2.50	..	..	..	Intended for normalized forgings.
Nickel-Chromium	0.25-0.45	0.40-0.80	2.75-3.50	0.60-0.95	..	..	Heat treated usually by quench and temper.
Nickel-Chromium	0.28-0.42	0.40-070	Min. 1.25	Min. 0.70	..	..	Slightly lower properties.
Nickel-Chromium	0.25-0.50	0.40-0.80	2.75-3.50	1.25-1.75	..	..	For higher tensile values.
Nickel-Chromium	0.20-0.45	0.40-0.80	Min. 4.50	1.25-1.75	..	..	"Krupp Analysis" for very high tensile values.
Nickel-Molybdenum	0.25-0.40	0.60-0.90	2.00-3.00	..	Min. 0.30	..	Suitable for normalizing or quenching.
Nickel-Chrome-Molybdenum	0.25-0.40	0.40-0.80	2.00-3.00	0.60-0.95	0.30-0.60	..	Suitable for normalizing or quenching.
Nickel-Vanadium	0.18-0.45	0.60-0.90	1.75-2.25	..	..	Min. 0.15	Suitable for normalizing or quenching.
Nickel-Chromium-Molybdenum-Vanadium	0.25-0.70	0.40-0.80	1.25-1.75	0.50-1.00	0.20-0.30	Min. 0.15	High values.
	Phosphorus and Sulphur usually specified under 0.045%						

Note: In the accompanying table of specifications, certain steels are designated "K", "L" and "M" types. This is in conformance with the specifications for carbon and alloy steel forgings of the American Society for Testing Materials (A18-27). Their analysis is not specified. The required physical properties can be obtained from any of the nickel-chromium or other compound steels given above.

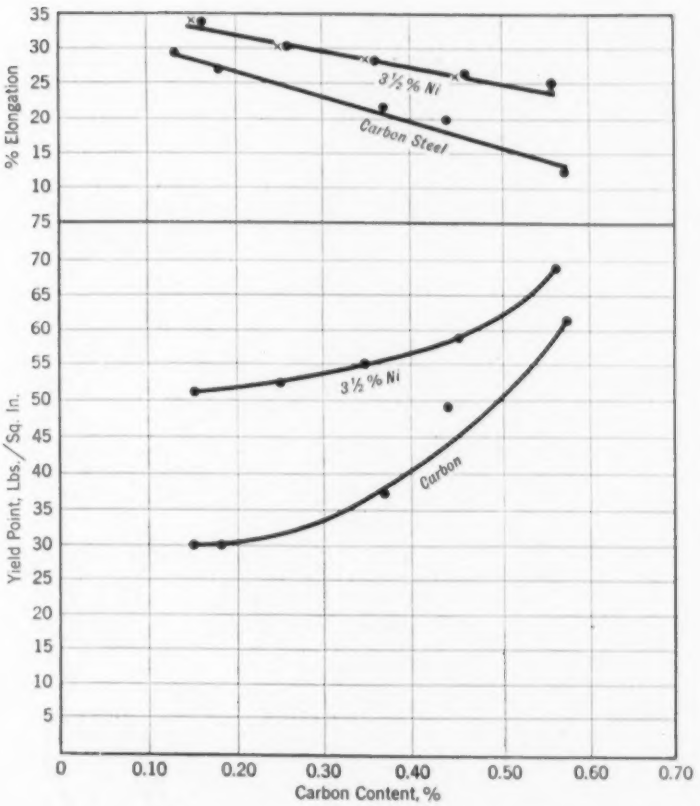


Fig. 2—Comparison of Normalized Nickel Steel and Carbon Steel Forgings.

It acts to improve the properties of steel principally through the formation of carbides. It is added to the steel bath as ferrochromium which carries other elements. It oxidizes during the process to some degree.

Molybdenum is very similar to chromium. It is more powerful than chromium in its effects, pound for pound, and in addition has the valuable characteristic of increasing the depth-hardening of steel and nullifying "temper brittleness." It acts for the most part through the formation of carbides. The addition to the steel bath is usually made in the form of calcium molybdate, from which pure molybdenum is reduced into the steel. Ferromolybdenum is also used. Like nickel, molybdenum does not oxidize and is recoverable from the scrap.

Vanadium acts as a toughener and strengthener of steel. It also forms carbides, but has the valuable property of reducing grain size. It oxidizes very readily and hence the addition of vanadium

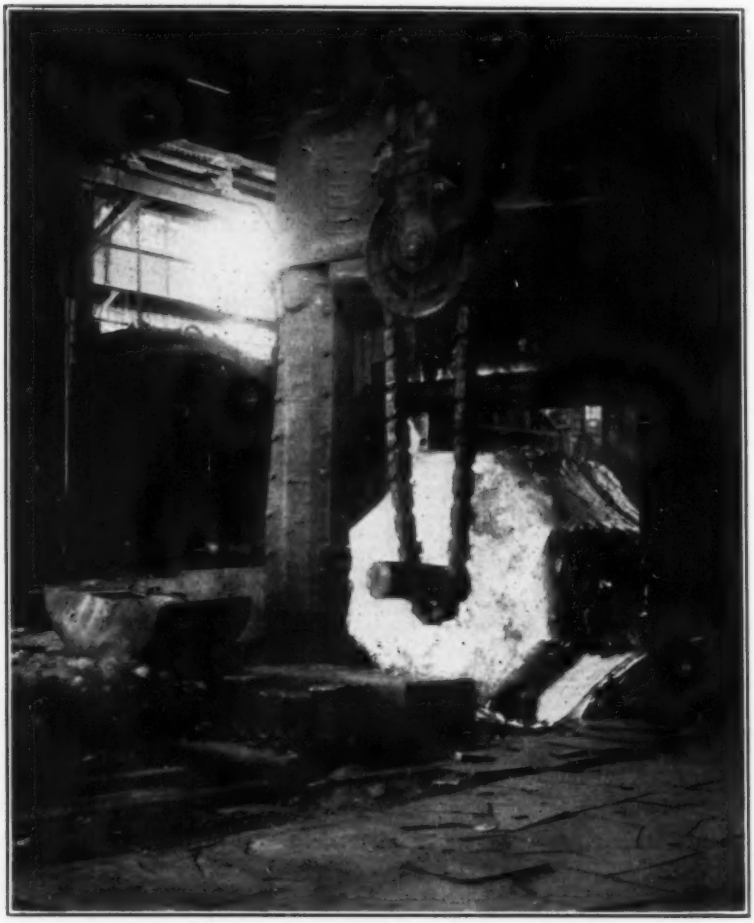


Fig. 3—Forging Large Ingot Under Hydraulic Press.—Courtesy Midvale Co.

in the form of ferrovanadium must be made after the steel is well-killed or it will be lost as an alloy.

Analyses

Nickel alloy steels for forgings comprise the following group. —Nickel steels; nickel-chromium, nickel-chromium-molybdenum; nickel-molybdenum; nickel-vanadium; nickel-chromium-vanadium; nickel-chromium-vanadium-molybdenum.

Nickel Steels: Steel containing 3.0 per cent nickel was the first alloy steel used in quantity for forgings, and has for years been a standard. Its use is indicated where carbon steels are inadequate and more strength, toughness and reliability required. It is used

either in the annealed, the quenched and tempered or the normalized condition.

While steel containing 5.0 per cent nickel is not much used in this country for large forgings, its qualities have made it much more popular abroad, where for all alloy steels it is customary to use a higher alloy ratio. The additional nickel gives a very strong steel with high dynamic strength which is equally well suited to any of the heat treatments. It also, in common with all straight nickel steels, gives very uniform results from heat to heat.

One of the interesting uses of straight nickel steels is that of using a nickel steel of low-carbon content in order to obtain great toughness and resistance to shock with only slight sacrifice of strength. Originally developed by one of our oldest forge companies for use in subway and street car axles, this steel contained only 0.13 per cent carbon and 3.0 per cent nickel, thus approaching a pure iron-nickel alloy. The heat treatment was the quench-and-temper, and it responded nicely, on account of the low carbon, with practically no danger of cracking. Subsequently it was used with excellent results for such parts as large stamp-mill shafts which were subjected to such severe impact that it was difficult to get any steel to stand up.

Recently the same steel, except with a slightly higher carbon and manganese, has been extensively used in the normalized condition for locomotive forgings, such as main and side rods, axles, piston rods and crankpins. For such service, its unusually high dynamic strength and toughness, together with its relative insensivity to heat-cracking makes it particularly suitable. Its toughness is definitely shown by Izod impact tests of from 50-70 foot-pounds in normalized forgings of fair size, which is about double that usually obtained from similar alloy steel forgings.

Nickel-Chromium Steels: By a combination of nickel and chromium it is possible to get a stronger, harder steel than by the use of either alone, yet one having good fatigue and impact values. Since these properties are best developed by the quench-and-temper heat treatment comparatively little nickel-chromium is used in the annealed condition.

It is commonly known that chromium steels exhibit under certain conditions a phenomenon known as "temper-" or "blue-brittleness" and nickel-chromium steels are no exceptions. If, for example, a nickel-chromium steel forging be slowly cooled from the tempering temperature it may be found that when cold the forgings

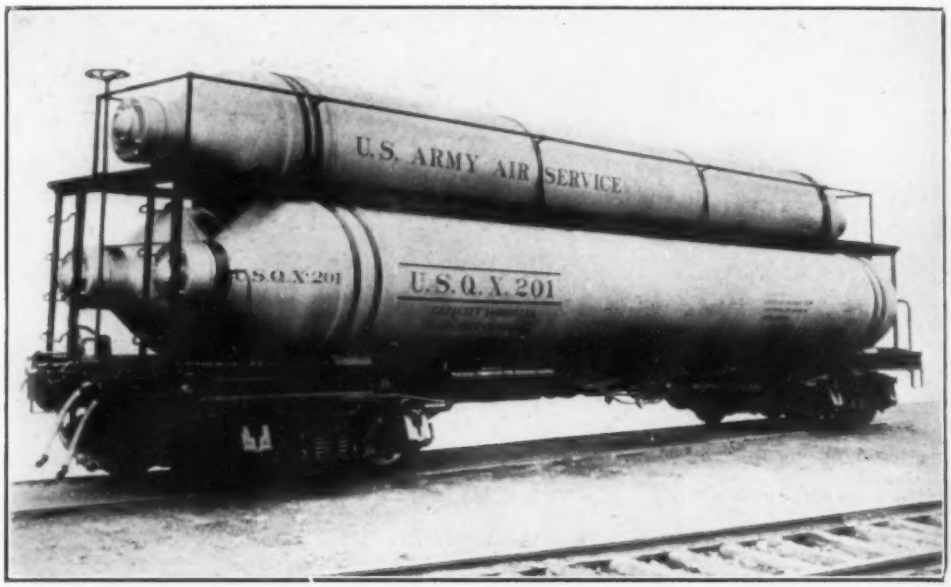


Fig. 4—Tank Car for Compressed Helium Forged Nickel Steel Cylinders.—Courtesy Bethlehem Steel Co.

are very brittle and will not withstand shock. The remedy is simple, —cool rapidly from the tempering treatment by quenching.

Nickel-Molybdenum Steels: The combination of nickel and molybdenum is an excellent one. Nickel-molybdenum steels are easier to make than steels containing oxidizable elements, they exhibit remarkably uniform properties, forge readily and respond nicely to any heat treatment. Molybdenum, in addition, has the property of eliminating temper-brittleness.

Nickel-Chromium-Molybdenum Steels: Although more expensive, a steel containing nickel, chromium and molybdenum has excellently balanced characteristics, being strong, tough and hard. It responds to heat treatment readily, and is free from temper-brittleness. This steel, or a similar one with the addition of vanadium, is quite widely used for die blocks, the large majority of which are made from nickel alloy steel.

Nickel-Vanadium and Nickel-Chromium Steels: These steels are not as extensively used as they might well be. Nickel-vanadium steel is used for large castings in order to obtain a fine-grained steel with high strength and ductility. The same features which make it valuable for castings recommend it for large forgings.

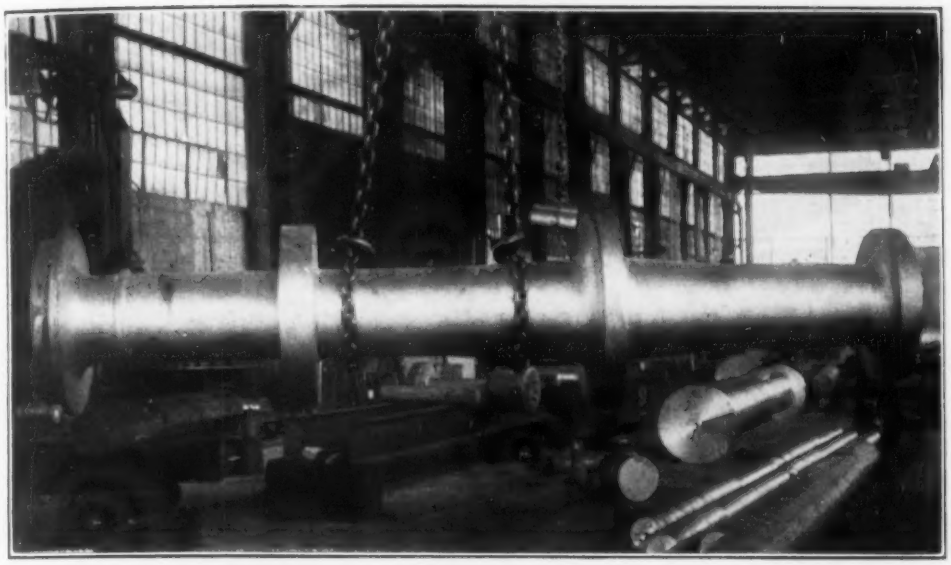


Fig. 5—Nickel Steel Flywheel Shaft Made of 3½ Per Cent Nickel Steel.—Courtesy Camden Forge Co.

Heat Treatment

All large forgings are, or should be, heat treated before delivery. The treatments used are the three standard ones of annealing, normalizing and quenching and tempering. Essentially, the procedure is the same as with smaller pieces, but it is imperative that consideration be given to the masses involved and stresses set up. Today annealing seems to be giving way to normalizing and the bulk of large forgings are either normalized or quenched and tempered. Further, for very large forgings, say forty inches in diameter and length or equivalent section, the normalizing treatment is generally the only one used. Few people realize the stresses set up in heat treating such a mass or the difficulties attending the operation. Illustrative is the comment of one manufacturer that large turbine forgings, rough bored axially to a six-inch diameter, will change size on the first of three heat treatments to such an extent that the bore will be only five inches in diameter.

The size of these forgings dictates the desirability of another change in the standard methods of heat treatment as applied to small pieces. In small forgings the grain is quite well-refined by the mechanical working and the ingot structure practically eliminated. This is not the case in large forgings and vestigial ingotism often persists after the forging operation. To break this up the heat

Specifications and Physical Properties Nickel Alloy Steel Forgings

			SPECIFICATIONS						REASONABLE EXPECTATION			
					Elongation in 2"		Reduction of Area					
Type	Treatment	Size	Tensile Strength	Elastic Limit	Min. % Inverse Ratio	Min. %	Inverse Ratio	Min. %	Tensile Strength	Elastic Limit	Elon-ga-tion	Red. of Area
Carbon	Annealed	up to 8" dia.	75,000	.5 Tens. Str.	1,800,000 / Tens. Str.	20.0	2,800,000 / Tens. Str.	33.0	75– 78,000	40–43,000	25.0	47.0
Carbon	Annealed	8"–12" dia.	75,000	.5 Tens. Str.	1,725,000	19.0	2,640,000	31.0				
Carbon	Annealed	12"–20" dia.	75,000	.5 Tens. Str.	1,650,000	18.0	2,400,000	29.0				
Carbon	Q & T	up to 4"–2" Max. Wall	90,000	55,000	2,100,000	20.5	4,000,000	39.0	90–95,000	55–60,000	23.0	45.0
Carbon	Q & T	4"–7" 3½" Max. Wall	85,000	50,000	2,000,000	20.5	3,800,000	39.0				
Carbon	Q & T	7"–10" 5" Max. Wall	85,000	50,000	1,900,000	19.5	3,600,000	37.0				
Carbon	Q & T	10"–20" 5"–8" Max. Wall	82,500	48,000	1,800,000	19.0	3,400,000	36.0				
Carbon	Normalized	10"–20" dia.	83,000	43,000	.....	22.0	.....	35.0	75– 85,000	40–45,000	23.0	43.0
3% Nickel	Annealed	up to 12" dia.	80,000	50,000	2,000,000	22.0	3,600,000	40.0	80– 90,000	50–55,000	25.0	47.0
3% Nickel	Annealed	12"–20" dia.	80,000	50,000	1,900,000	21.0	3,400,000	38.0				
Low Carbon 2.5% Nickel	Normalized	10"–20" dia.	80,000	55,000	.....	27.0	.....	50.0	80– 90,000	57–65,000	28.0	52.0
3% Nickel	Q & T	up to 4"–2" Max. Wall	100,000	70,000	2,200,000	20.0	4,500,000	41.0	100–110,000	70–80,000	23.0	45.0
3% Nickel	Q & T	4"–7" 3½" Max. Wall	100,000	65,000	2,100,000	20.0	4,300,000	41.0				
3% Nickel	Q & T	7"–10" 5" Max. Wall	90,000	60,000	2,000,000	20.0	4,100,000	41.0				
3% Nickel	Q & T	10"–20" 5"–8" Max. Wall	85,000	55,000	1,900,000	20.0	3,900,000	41.0				
3% Nickel	Normalized	10"–20" dia.	90,000	60,000	.....	22.0	.....	40.0	90– 95,000	60–65,000	25.0	45.0
Ni.-Cr. Steel Type K	Q & T	4"–7" dia. 3½" Max. Wall	90–110,000	65,000	.....	20.0	.....	50.0				
Ni.-Cr. Steel Type K	Q & T	7"–10" dia. 5" Max. Wall	90–110,000	65,000	.....	20.0	.....	50.0				
Ni.-Cr. Steel Type K	Q & T	10"–20" dia. 5–8" Max. Wall	85–105,000	60,000	.....	20.0	.....	50.0				
Ni.-Cr. Steel Type L	Q & T	4"–7" dia. 3½" Max. Wall	100–120,000	75,000	.....	20.0	.....	50.0				
Ni.-Cr. Steel Type L	Q & T	7"–10" dia. 5" Max. Wall	100–120,000	75,000	.....	18.0	.....	45.0				
Ni.-Cr. Steel Type L	Q & T	10"–20" dia. 5–8" Max. Wall	95–115,000	70,000	.....	18.0	.....	45.0				
Ni.-Cr. Steel Type M	Q & T	4"–7" dia. 3½" Max. Wall	110,000	85,000	.....	16.0	.....	45.0				
Ni.-Cr. Steel Type M	Q & T	7"–10" dia. 5" Max. Wall	100,000	75,000	.....	18.0	.....	45.0				
Ni.-Cr. Steel Type M	Q & T	10"–20" dia. 5–8" Max. Wall	100,000	70,000	.....	18.0	.....	45.0				

treater often resorts to a high temperature normalizing treatment prior to the treatments designed to improve the physical properties. This treatment is usually between 1800 and 2000 degrees Fahr. and of long duration.

Nickel, it is well-known, lowers the critical range of the steel to which it is added. This is definitely advantageous as there is less liability to increased grain growth at the lower temperatures and also less liability to warpage and internal stresses. Nickel steels also are not injured, but improved, by long soaking in the critical range.

Whenever possible, forgings should be axially bored. There is a fourfold reason for this. The physical properties are benefited by the increased rate of cooling; the material at the axis is not particularly valuable from an engineering standpoint as the stresses are lowest here; the metal which is inherently the poorest is removed and the resulting bore affords an opportunity for internal inspection which is very desirable.

Properties

To venture into the field of what properties may be expected and what properties should be specified for forgings is to tread on dangerous ground. Smaller objects are more uniform in size, are more homogeneous and respond more uniformly to heat treatments. Each large forging is a law unto itself. Large forgings vary in simplicity and in size, and to attempt roughly to classify them by size necessarily results in generalization. It is somewhat as if one attempted to write a general specification under which all makes of automobiles would be included. To illustrate this point, Fig. 1 shows two "heavy forgings". One is a simple cylinder, bored axially, and of comparatively small mass. The other is of great mass with extreme and rapid variations in section and shape. Yet both come under the general specification of "large forgings".

The result of such enforced conservatism is, happily, that the purchaser may reasonably expect higher properties than are guaranteed, unless his forgings are extreme in size or intricacy. The attached table gives for large forgings of nickel alloy steel specifications which are considered standard, the properties which may reasonably be expected from what might be called the "average" of each group and concrete examples of properties which have actually been obtained in practice.

Ni.-Cr. Steel Type M	Q & T	7"–10" dia. 5" Max. Wall	100,000	75,000	.....	18.0	..... 45.0
Ni.-Cr. Steel Type M	Q & T	10"–20" dia. 5–8" Max. Wall	100,000	70,000	.....	18.0	..... 45.0

Inspection

This paper would be incomplete without a word on inspection—perhaps the most important and certainly the most damned function connected with all steel production. Tensile test pieces intelligently chosen give a good idea of the character of the object inspected. Unwisely picked, they are worse than useless. Test pieces from some forgings for locomotive main rods were to be taken from a "prolong in the end of each forging." Since the size of the prolong was not specified, in this case they were reduced to a 1½ each square on the end of forging some 8 x 14 inches. Obviously, some one was fooled.

The heavy etching of blooms for forgings and for forgings themselves has come into vogue recently. This, it seems, can only tend toward the betterment of the product and therefore advantageous to both the purchaser and the manufacturer. The fly in the ointment is that of interpretation of results, and it is very difficult to set standards of what is and what is not acceptable. The same is true of the microphotographic standard included now in many specifications. Unquestionably of great value if intelligently interpreted, it may become simply an onus to the conscientious producer.

Conclusion

The quality of large forgings has been getting progressively better for many years. Today the designing engineer can justifiedly have entire confidence in material of this sort. But, through the use of improved steel-making processes, the further use of alloys and the improvement in mechanical and heat treatment it is entirely probable that the product a few years from now will be as much better than the product of today as the product of today is better than that of twenty years ago.

DISCUSSION

Glenn Coley:[1] I would like to ask Mr. McKnight what treatment he would suggest to remove banded structures.

Charles McKnight: That is a hard question to answer. I would be inclined to use a high normalizing treatment, but that is not always successful. No heat treatment is always successful in breaking up banded structures, but you will get the best results from a heat treatment of 1800 to 2000 degrees Fahr. normalizing, and air quenching from that.

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[1]Member A. S. S. T., power sales department, Detroit Edison Co., Detroit.

CHAIRMAN E. C. BAIN:[2] You do not know of any other more efficacious method?

CHARLES MCKNIGHT: No.

ROBERT JOB:[3] I should like to ask Mr. McKnight what influence nickel and other alloys have upon locomotive driving axles as regards the tendency to form heat checks.

CHARLES MCKNIGHT: That is a very embarrassing question to ask. Most of the trouble with locomotive driving axles has been one of lubrication. The solution to the locomotive axle difficulty, as I see it, is not a metallurgical solution, but a mechanical solution. Eventually, we will have to adopt the European practice of forced lubrication. With the present steels you are going to run up against the question of heat cracking. Some steels are better than others. As a general rule, the lower the carbon, the better the steel is for resisting the heat cracking. I do not know that nickel is any better than any other alloy as far as heat cracking is concerned, but it does work well in low carbon steel. On tests that have been run with various alloy steels where the axle has been run until it got to a red heat, and one end was allowed to cool naturally and the other end was quenched in water, the low carbon nickel steel showed up better than the other steels.

ROBERT JOB: My object in asking the question was to bring out the point which Mr. McKnight raised. I have heard a statement made that one alloy or another would have a decided influence upon the tendency to form checks. I believe, as Mr. McKnight does, that the question of lubrication has a great deal to do with it. It is very important on that account to consider the method of lubrication. I was particularly interested to know under what conditions nickel was recommended, also what proportion of carbon would best be present in order to avoid tendency toward heat checks. To a certain extent, we have to take conditions as we find them. Assuming that we get a certain kind of lubrication with the harder type of lubricant, I was wondering whether Mr. McKnight would consider it desirable to use the lower range of carbon if nickel steel were used. I think that whether you use nickel or some other alloy would be immaterial, to a certain extent, but it may be that a lower range of carbon would be desirable in order to avoid the tendency toward the heat checking if the harder range of lubricant is used.

CHARLES MCKNIGHT: Nickel is about the only alloy that will give you alloy steel figures with low carbon. I think that the eventual answer on the driving axles will be better lubrication and heat treated steel.

W. J. MERTEN:[4] I have several points on which I want to raise questions. Mr. McKnight mentions 4-inch diameter forgings as a starting point. Naturally my category of forgings is large forgings. In the heavier forging types of a minimum of 30-inch diameter up to 80 inches diameter or more, of some hundred thousand pounds of weight, it is generally accepted that the acid open-hearth process is the melting process employed for a quality forging. I believe

[2]Member A. S. S. T., research staff, U. S. Steel Corp., Kearny, N. J.

[3]Member A. S. S. T., vice-president, Milton Hersey Co., Ltd., Montreal, Canada.

[4]Member A. S. S. T., metallurgical engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh.

the speaker had this in mind when he mentioned really large forging melting practice.

Mr. McKnight also mentioned that a press forging is of a better quality than a hammer forging. From my observations I want to say that a press forging is a much more difficult forging to make, and it takes considerably more skill to produce a forging of high quality, than under a hammer. A hammer forging is probably safer to produce than a press forging because the skillful operation of a press is not an every day man's experience.

I would also like to ask how much molybdenum is necessary to reduce the temper brittleness of a chromium-nickel material. I have my doubts that larger sizes, particularly those that are not quenched, will respond to a reduction. Unless grain refinement is complete, I doubt that temper brittleness is removed.

Mr. McKnight also states that the addition of nickel to low carbon steel results in a slight sacrifice of strength. I do not think he means that. I have always considered nickel as a strength-inducing element. Lowering of tensile strength, to get high impact values, is bad practice on nickel steels as well as on plain carbon steels.

Mr. McKnight mentioned the simple method of quenching a forging to temper brittleness. To quench a large ring forging from a tempering heat and keep its form without warping is quite difficult. In addition internal stresses are introduced seriously affecting the practical application of such forging. It is not simple to avoid temper brittleness.

The vanadium-nickel type of casting is another subject which I would like to dwell upon for a few minutes. I have had some experience with castings of this type, and, in order to get the benefit of the alloying elements, we had to go to 2200 degrees Fahr. to bring about uniform grain refinement and proper conditions to justify the increased cost over that of other alloy steel. Even in small forgings there is a large amount of segregation occurring which is defeating the use of higher stresses in service from castings of this type.

CHARLES MCKNIGHT: With regard to the amount of molybdenum it takes to produce temper brittleness, I want to pass the buck to some molybdenum producer. I would say that 0.15 or 0.20 per cent would reduce it. In England where they use a relatively large amount of nickel-chromium-molybdenum steel with all kinds of heat treatment; i. e., annealing, normalizing and quenching, they claim that molybdenum eliminates the liability of temper brittleness. I have to agree with Mr. Merten that it is not simple to cure temper brittleness in a large forging. I think I mentioned the fact that it was very difficult to heat treat large forgings.

My thoughts were worded badly when I spoke of a slight sacrifice of tensile strength with low-carbon steel. Certainly the addition of nickel does result, as Mr. Merten says, in strengthening this material. What I meant was that in the railroad field where they had been using a normalized alloy steel of fairly high tensile strength, say 90,000 pounds per square inch, they have been able, by substituting a low-carbon nickel steel with an ultimate tensile strength of 75,000 to 85,000 pounds, to get longer life and less breakage.

W. H. PHILLIPS:[5] I have been very much interested in the fine paper that

[5]Member A. S. S. T., vice-president, Molybdenum Corp. of America, Pittsburgh.

Mr. McKnight has presented. If I understand Mr. McKnight correctly as to the tendency of molybdenum to form carbides, we believe that in the small quantities usually required in structural steels, namely up to ½ of 1 per cent, all of the molybdenum will be found in solution with the iron. The formation of carbides would lessen the tendency of molybdenum to reduce temper brittleness. Mr. Merten and I have had several arguments on the effect of molybdenum on chromium-nickel steels in its tendency to eliminate temper brittleness. He believes that the nickel-molybdenum steels show everything that can be secured with chromium-nickel-molybdenum steels and they have no tendency to form temper brittleness.

The Research Department, Woolwich, London, in their report No. 67, state page 12: "The chromium and nickel-chromium steels were slightly, but distinctly, susceptible to temper brittleness, but in every case steels containing 0.30 per cent molybdenum and over showed no signs of temper brittleness". And again, page 20: "molybdenum has a pronounced effect in reducing, or in some cases completely eliminating the susceptibility of the steel to temper brittleness." "No rate of slow cooling from the tempering temperature likely to be met with in practice has any effect in reducing the impact figure of steels of the types investigated containing more than 0.30 per cent of molybdenum."

I believe that molybdenum contents of from 0.20 per cent to 0.30 per cent molybdenum will have a pronounced tendency to reduce or eliminate temper brittleness.

GLENN COLEY: Mr. Merten mentioned the fact that he thought hammer forgings were superior to press forgings. I would like to take up that on large sections, such as Mr. McKnight has been discussing. I do not believe it possible to get the working throughout the section on a hammered forging that can be obtained with a pressed forging.

W. J. MERTEN: If I have not stated my thoughts correctly, I will try to do so now. I mean to say that it takes a great deal more skill to press right than to hammer right, and a hammer forging obviously is better on small forgings for economical reasons.

A. H. D'ARCAMBAL:[6] Mr. McKnight mentioned several different nickel alloy steels, such as nickel-chromium, nickel-vanadium, etc. In our plant at Pratt and Whitney we machine several different parts for airplane engines made of alloy steel heat treated from 250 to 300 Brinell. We would be interested in learning the type of alloy steel heat treated to that Brinell that has the best machining properties including chromium-vanadium steels.

W. J. MERTEN: From a standpoint of machinability I think the chromium-nickel steels are better than the chromium-vanadium steels. However, again, I have to boost the molybdenum. In a nickel-molybdenum steel, even a higher degree of hardness gives machinability which is very much better than with other alloys, that we have encountered, at lower hardness from a standpoint of uniformity of cutting qualities as well as cutting efficiency.

CHAIRMAN E. C. BAIN: I was thinking that if the fairly abundant evidence that we have in these days is all correct, that machinability has come in very close connection with the structure, the distribution, and nature of the

[6]Member A. S. S. T., consulting metallurgist, Pratt and Whitney Co., Hartford, Conn.

carbides, so close that I would ask Mr. d'Arcambal to answer his own question in the light of structures obtained.

A. H. D'ARCAMBAL: In a large number of the aircraft parts referred to, we are using number 6150 steel, heat treated from 250 to 300 Brinell. This material has the usual fine grain structure you obtain through proper treatment. However, in some of the heat treated die blocks you will notice that they add molybdenum to their alloy steels. It seems to be the general consensus of opinion that when you come to the high Brinell material, 300 Brinell and higher, molybdenum must be present to give you a machining operation. I think Mr. Phillips will agree to that.

H. B. KNOWLTON:[7] With regard to the question of machinability I might say that we have used nickel-chromium and nickel-chromium-molybdenum steel. The claim was made that the latter would give the best machinability in proportion to Brinell hardness, but due to the difference of opinion among different machine operators, I cannot state which was the more machinable. We did find in the case of the nickel-chromium-molybdenum steel a greater penetration of hardness and a higher practical working strength in proportion to surface Brinell. That is, a tensile test bar taken half way between center and outside showed better figures. The gain was not great, but was sufficient to be of commercial value.

I would like to ask the speaker to what degree of brittleness he was referring in his statement that nickel-chromium steel was brittle if not quenched after the draw. I think perhaps he was referring to a paper published in England a few years ago which showed a slight difference between the properties of nickel-chromium and chromium-molybdenum steels when slowly cooled from the tempering treatment, in favor of the latter steel. I do not recall the figures and wonder if the speaker can tell us how great the difference is. I agree with Mr. Merten that quenching from the tempering temperature is not always so desirable.

CHARLES MCKNIGHT: Again you will have to fall back on some of the consumers to give a reply to that question.

W. J. MERTEN: May I relate an experience I had on temper brittleness which is a rather peculiar one. My friend, Mr. Phillips, was particularly interested in this question at one time on small gears for starting motors. These were all 1 per cent chromium and 3 per cent nickel steels, and were tempered at around 600 degrees to obtain a hardness of around 375 to 400 Brinell, and every pinion taken from the furnace when struck with a hammer, went to pieces. By quenching these pinions from the tempering heat, temper brittleness was removed, and on small parts of this type quenching is the remedy and cure.

H. J. FRENCH:[8] Mr. Knowlton is quite right when he says it is difficult to evalute "machinability" and the different criteria employed at different times in discussing the machining characteristcs of metals sometmes have made it difficult to reconcile what appear to be anomalous or contradictory results. However, a few general principles should be recognized in any discussion of the subject.

[7]Member A. S. S. T., assistant chief metallurgist, International Harvester Co., Chicago.

[8]Member A. S. S. T., research laboratories, International Nickel Co., Bayonne, N. J.

The ease of machining of difficult steels is first a function of the nature of the cutting operation (turning, drilling, etc.) and, secondly, the manner in which they are carried out (form and character of tools, cutting liquids, cutting speeds, feeds, etc.). But even with standardized cutting conditions in a given operation the superiority of one steel over another, in the way in which it can be machined, is not solely a function of the chemical composition of the steel as now ordinarily determined. Variations in grain size, indicative of variations in methods of manufacture, must also be considered, and it is known that steels with good machining characteristics at low hardnesses do not necessarily show superiority when heat treated for somewhat higher hardnesses.

In regard to impact tests a method of detecting temper brittleness is to compare the notch test values on samples slowly cooled from the tempering temperature with those rapidly cooled (quenched in water). Temper brittleness is indicated when the notch test values of the slowly cooled samples are appreciably lower than those of the rapidly cooled samples.

CHAIRMAN E. C. BAIN: Thank you, Mr. French. I take it that while you are pretty well established with constant tool quality, the rough turning follows it reasonably close to the tensile strength. That might not be true for face finishing, in which the surface left on the work is of extreme importance, and you are not interested in removing large amounts of metal.

G. J. SAWYER:[9] I would like to ask Mr. McKnight if he has had any experience in retaining austenite in 5 per cent nickel steels on the quench when using salt as a heating medium. Also if he has ever experienced any cracking in 40 carbon chromium-nickel-molybdenum steels having key ways or small sections in quenching out from the salt medium.

CHARLES MCKNIGHT: In answer to the first question, I have had no experience along that line, but as regard the cracking of the 40 carbon chromium-nickel-molybdenum steel from the salt bath, the key way, I think, is of vast importance. As far as the austenite is concerned, we have an asset in Mr. French. Will you answer that question, Mr. French?

H. J. FRENCH: Soft cases in case hardened alloy steels are frequently due to austenite retention. One practical method of eliminating this difficulty is to lower the maximum carbon concentration of the case, by compound dilution or other means, and harden the carburized steel from the lowest practicable temperatures.

G. J. SAWYER: I am able to get away with it by going to open fire and forming a scale, but the minute I go to a salt bath I seem to retain it or else drop the temperature way down, which will give a bad core.

W. J. MERTEN: May I make a suggestion? I think we have a man in the audience with a large amount of experience on a 5 per cent nickel alloy and that is Mr. Mochel.

N. L. MOCHEL:[10] I had not intended to get into this discussion, but now Mr. Merten has me in it. It so happens that I have supervised the working of several millions of pounds of 5 per cent nickel steels in the last few years. The

[9]Member A. S. S. T., supervisor of steels, Cleveland Tractor Co., Cleveland.

[10]Member A. S. S. T., metallurgist, Westinghouse Electric and Manufacturing Co., Philadelphia.

question about austenite affected us in a little different way than it has the last speaker. I understand he is referring to case hardening work. We have done some work relative to case hardening, but not a great deal. Our experience has been with drop forgings of this material, for turbine blades. Our standard method of heat treating had always been to quench in oil and temper, a fairly high temper, around 1200 degrees Fahr. We have found in a number of cases that we had what we have always felt was retained austenite from this oil quench. We would have some heats of steel that would behave all right. We would find an occasional heat that would not behave normally to the quench. We have found that we could get the desired Brinell hardness and the tensile strength by reverting to water quenching. It is true that one cannot always go to water quenching. It happened in our case that we could, and we found that if we quenched in water at a temperature of 100 degrees Fahr., that we could get the Brinell hardness we desired. I think there is much in the work which has been done by Dr. Mathews and others to show that the quenching of alloy steels in oil will result in the retention of considerable austenite, whereas if you quench in water you do not get it. I do not know whether this adds much to the discussion or not, but that has been our experience.

SCALING OF STEEL AT FORGING TEMPERATURES

By W. E. Jominy and D. W. Murphy

Abstract

The relative scaling action of air, steam, carbon dioxide and oxygen on steel at 2000 to 2500 degrees Fahr. is presented in this paper. In addition, the effect on scaling of time of exposure, of rate of flow of the scale producing gas, and of variation of temperature is given. It is shown that the rate of scaling decreases with the time of exposure and increases with the temperature. Of special interest is the finding that increasing the rate of flow of the gas above 50 feet per minute has practically no effect on the rate of scaling. Between zero and 10 feet per minute, the rate of flow has a very pronounced effect, the slower the rate, the less the scaling.

THE factors involved in heating steel for forging do not seem to have received the same thought and consideration that have been given other steel-heating processes. As a result improvement in methods of heating steel for forging has not been comparable with that in the heating for heat treating. Automatic temperature control, and continuous furnaces are now quite general in heat treating work and it is quite uncommon to find heat treatment without the use of pyrometers. In heating for forging purposes, however, it is almost as uncommon to find thermocouples employed in temperature measurement. It is possible, of course, to use a wider range of temperature in forging than in heat treating, but there are advantages to be gained by uniformity of heating and by forging at the higher temperatures. Under these conditions, to be sure, it is necessary to maintain temperature control, if burning and overheating are to be avoided, but forging is easier to perform, and there is greater freedom from defects due to the easier flow

This paper is a study made as part of the program of investigation of the various metallurgical factors involved in heating steel for forging which is being conducted at the University of Michigan for the American Gas Association.

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of the steel at the higher temperatures. The temperatures at which some of the more common steels will burn and the conditions under which burning may be expected or may be avoided have been studied as part of a program of investigation in process at the University of Michigan and have been published in a previous paper.[1]

One of the metallurgical factors involved in heating steel for forging is the scaling of the steel. This scaling, which is more or less taken for granted in any forge shop, is really more of a factor than is usually realized. It is not always appreciated that scaling enters definitely into a number of items of cost other than the mere loss of metal and, further, that scaling is an important cause of a number of undesirable qualities in the finished forging. A consideration of the following facts will give some idea of the difficulties resulting from the presence of scale and of the added costs of a finished forging due to scaling:

1. Scale from steel reacts with most refractories to form a relatively low melting compound. This reaction greatly shortens the life of furnace floors and other refractories with which the scale may come in contact.

2. Scale from the steel occupies space in the forging dies, and results in parts with wrong dimensions. It can be and often is scraped off before forging, if excessive, but this process requires additional labor.

3. Scale formed on steel causes a loss in the weight of steel, and therefore causes an economic loss equal to the value of the steel transformed into scale.

4. Scale on steel sometimes causes sticking of the forging in the forging dies. This, of course, causes increased labor cost.

5. Scale on steel sometimes gets pounded into the forging, causing weak spots in the forging and points at which cracks develop easily. This embedded scale also sometimes causes machining difficulties.

6. Most forgings must have the scale removed before the forging is machined or used. This removal of the scale, whether by pickling, tumbling, or shot blasting, is more expensive the deeper and more adherent the scale.

It is evident that the presence of scale in a finished drop forg-

[1]W. E. Jominy, TRANSACTIONS, American Society for Steel Treating, Vol. 16, 1929, p. 298, 372, 893.

ing which results from the action of the atmosphere in the forging furnace seriously affects its quality and adds considerably to its cost. The investigation which forms the subject of this paper was consequently undertaken in order to make a study of the phenomena of scaling of steel at forging temperatures in the hope of throwing some new light on the principles involved with the ultimate objective of improving the method of heating forgings.

Previous Work

No published previous work has apparently been carried out on scaling at temperatures around 2300 degrees Fahr. Marson and Cobb[2] carried out tests on the scaling action of air, water vapor and carbon dioxide at temperatures of 1652 to 1832 degrees Fahr. Their experiments appear to have been carefully made so far as purity of gases used and method of measuring the scale formed is concerned. They do not state anything, however, regarding the velocity of the gas as it passes the sample except that the rate of flow was uniformly from 2½ to 3 liters per hour. According to the findings of the present research, the rate of flow of gas past the sample has a very pronounced effect on the rate of scaling, particularly if the rate of flow is near zero. Through correspondence with C. B. Marson, it was learned that the size of tube employed in his research was two inches. This would result in a rate of flow of gas in the tube of 0.425 feet per minute, which is in the range where small changes in rate of flow have a pronounced effect on the rate of scaling. It would be expected, therefore, that, although these results may be fairly comparable, they cannot be applied at rates of flow appreciably slower or faster. This has a limiting effect on the possible application of these data.

Hatfield[3] reported the effect of air, carbon dioxide, and steam on pure iron and on low carbon steel at temperatures from 1292 to 1832 degrees Fahr. Hatfield, however, also failed to mention the rate of flow of the gases. Dickensen,[4] and McCormick[5] have

[2]C. B. Marson and J. W. Cobb, *Journal*, Society of Chemical Industry, Vol. 46, 1927, p. T61.

[3]W. H. Hatfield, *Journal*, Iron and Steel Institute, No. 1, 1927, p. 483.

[4]J. H. S. Dickenson, *Journal*, Iron and Steel Institute, No. 2, 1922, p. 103.

[5]G. C. McCormick, Transactions, American Society for Steel Treating, Vol. 2, 1922, p. 1006.

likewise made studies of the rate of scaling, but have not mentioned the rate of flow of the gases. Pfeil[6] made a thorough study of the mechanism of scaling with air. He presented a hypothesis involving the diffusion of oxygen through the scale to the metal, and of iron outward from the metal into the scale. Pilling and Bedworth[7] also postulated a mechanism of scaling which involved a solution and diffusion of oxygen through the scale but did not include the outward diffusion of metal.

Method of Testing

In deciding on the method of approach for the present study some thought was first given to the use of equilibria diagrams. A great deal of work has been done in the study of blast furnace reactions and equilibria diagrams have been obtained of the various systems: iron, iron oxide, carbon dioxide, and carbon monoxide; and iron, iron oxide, water vapor and hydrogen. Although some idea of general tendencies could be obtained from a consideration of equilibria constants and free energy, these data could not generally be used, because, in the first place, the diagrams would have to be extrapolated in order to obtain data at forging temperatures, and secondly, the scaling reaction that occurs in a forging furnace never goes to completion and the study is principally one of rate of reaction rather than of results at equilibrium. For these reasons, it was decided to make tests by direct trial under chosen conditions.

In order to make such a study of scaling of steel it was deemed advisable to study first the simple, common gases that may be present in the atmosphere of a forging furnace, since with the simple gases it would be easier to discover the principles involved in the scaling phenomena. Consequently, the study which is reported in this paper was limited to the action of the gases: carbon dioxide, steam, oxygen, and air on steel at forging temperatures. The steel used in the test was 13/16-inch round S.A.E. 1015 steel. The temperatures at which tests were made varied from 2000 to 2500 degrees Fahr., and the periods of exposure varied from 5 to 160 minutes.

In conducting the tests a sample of steel was placed in a tube

[6]L. B. Pfeil, *Journal*, Iron and Steel Institute, Vol. 119, 1929, p. 501.

[7]N. B. Pilling and R. E. Bedworth, *Journal*, Institute of Metals, Vol. 29, 1923, p. 529.

through which the gas to be investigated was passing at a given rate and which had been previously heated to the desired temperature. The steel was allowed to be exposed to the conditions of tests for a predetermined period of time, after which the steel was removed from the furnace and dropped into cold distilled water to stop the reaction quickly and to split off part of the scale. The remainder of the scale was removed by electrolytic pickling. The steel sample was carefully weighed on an analytical balance before it was placed in the furnace and again after the scale was removed. From the difference in weight the loss of steel per 100 square inches of surface exposed was calculated.

Before any scaling tests could be made, it was necessary to develop a method for removing the scale from the steel. This has always been a somewhat perplexing problem in the study of the scaling of steel. A number of methods were tried out and the one which proved most satisfactory consisted in quenching the steel in water at the end of the heating which not only split off most of the scale but also stopped the scaling reaction quickly. However, all of the scale does not come off when the sample is quenched in water so that the sample must be treated further in order to remove the remainder of the scale. The removal of this remaining scale was found to be best accomplished by means of an electrolytic pickling bath, using a lead anode and making the specimen the cathode, using a four per cent solution of sulphuric acid in water and a potential difference of three volts. A blank of similar size and surface was immersed in the same solution with the specimen and carefully weighed before and after the pickling action. The sample was pickled in this solution for a period of time from fifteen minutes to three hours, depending on the type of scale, the average time being about forty-five minutes. The blank placed in the solution with the steel seldom lost more than two milligrams in weight indicating that the amount of steel dissolved in the pickling solution was practically negligible.

The scale was filtered from the distilled water into which the sample was quenched and weighed. To this weight was added the scale which came off in the electrolytic pickling bath. The scale which dissolved in this bath was then also determined. By adding these three values, it was possible to determine the weight of scale removed which could be compared with the weight of iron lost from the same sample.

The method of quenching in water is open to some slight objection due to the reaction of the steel at forging temperatures with the water. This reaction is known to produce scale, although, since the time of cooling is very short, the amount of scale produced is small. Another disadvantage is that the steel sample travels through air for a fraction of a second before it becomes immersed in the water. For these reasons, a few tests were made in which the steel sample was quenched after heating in an atmosphere that would produce no scale and the amount of scale found on the steel after quenching was measured. This was found to be about 0.003 pound per 100 square inches of surface. However, when the sample is coated with scale, as it was in all the tests, this scaling due to brief exposure to air and water is probably very much less.

Description of Furnace

The furnace used for the tests consisted of a gas-fired muffle furnace through which was placed a porcelain tube with glazed finish on the outside. Fig. 1 shows a diagrammatic sketch of the furnace and furnace equipment. The porcelain tube was found by test to be quite impervious to the passage of the products of combustion from the heating chamber of the furnace and the corundum muffle, although permitting these gases to filter through easily, produced fairly uniform temperature along the tube. The tube was stopped with a firebrick plug at one end which was cemented in place. Through this plug the thermocouple tube and the gas inlet tube were run, care being taken to cement these tubes in place. The other end of the tube was stopped with a plug, as shown at J, which had to be removable in order to facilitate placing the sample in the furnace and pulling it out. This plug was made of firebrick wound with asbestos tubing so that a fairly soft surface was obtained on the plug, permitting a reasonably tight joint. The joint, however, was not gas-tight, but, since the gases were always flowing in the direction of the plug, except in the cases of the still runs, a difference of pressure was maintained which prevented the infiltration of air. In the case of the still runs, the plug at this end was sealed in with cement after the sample was put in the furnace, after which test this cement seal was broken and the sample removed. The sample was mounted on a chromel-alumel wire boat so designed that the steel sample did not touch the refractory at any time and so that

the steel sample was at a height in the tube as shown in the sketch. The longitudinal placing of the sample in the tube was such that the bead of the thermocouple was at about the middle of the steel sample and about ¼ to ½ of an inch from the sample.

Before making a test, the gas to be used was always permitted to flow through the tube for a period of about twenty minutes, after which an analysis was made of the gas coming from the tube in order to determine whether the air in the tube had been

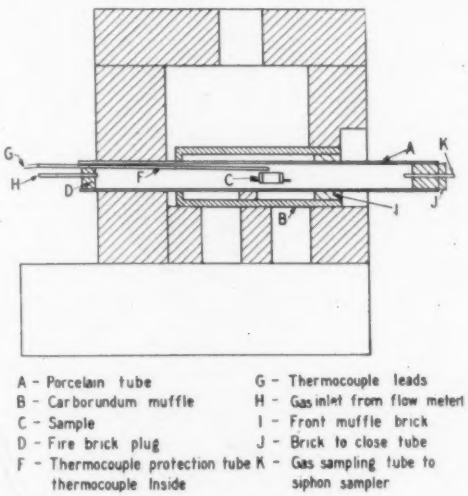


Fig. 1—Diagram of Gas Furnace Used in Scaling Tests.

washed out by this process. If no oxygen was found in the test, the gas was considered sufficiently pure for use in the conduct of the test. The gas analyses were made with a modified Williams Orsat gas analysis equipment, the modification of the equipment being a substitution of a sample burette which could be read more accurately than the usual Orsat equipment.

The measurement of temperature was by means of a platinum, platinum-rhodium thermocouple with a suitable refractory protection tube, and a potentiometer. The temperature readings were made every minute during the time of testing and variations from the average temperature were within ± 5 degrees Fahr. The period of time the sample was in the furnace was counted by means of a stop watch from the instant the sample was placed in the furnace until it was removed. The rate of flow of gas was measured by means of a mercury flow gage calibrated against a wet test meter.

The steel sample was prepared in every case by polishing it

with No. 00 emery cloth till the surface was perfectly bright. Care was exercised not to touch the sample with the hands once the surface was clean and bright. Samples were kept in a desiccator until they were mounted on the wire boat previous to placing in the furnace.

Results of Tests

Tests were made to determine the scaling effect of air, oxygen, carbon dioxide, and steam. In the case of the air, carbon dioxide, and steam, tests were made to determine (a) the rate of flow of the gas on scaling of steel at 2300 degrees Fahr., (b) the effect of the variation of temperature from 2000 to 2500 degrees Fahr., (c) the effect of variation of time of exposure of the steel at 2300 degrees Fahr.

The steel used consisted of a bar two inches long and 13/16 inches round, weighing approximately 130 grams, cut from bars of steel of the following composition: Carbon, 0.13 to 0.15 per cent; Manganese, 0.49 per cent; Sulphur, 0.029 per cent; Phosphorus, 0.018 per cent; Silicon, 0.14 per cent.

Air

Tests were first made with ordinary undried air in order to have the same conditions as when steel is heated in a muffle furnace, and difficulty was immediately encountered in obtaining satisfactory check results. Good check results were obtained when the tests were made immediately following one another, but tests made on another day under seemingly identical conditions, although checking each other, did not check the runs of the previous day. These discrepancies, it was felt, might be due to the varying moisture content in the air and, for this reason, tests were made with air which was first passed through two large U tubes containing calcium chloride to remove the moisture. With the moisture removed, it was possible to obtain fair checks with tests made even at intervals of several days or weeks. There still is, however, a tendency to obtain very good checks in tests immediately following one another and only fair checks in tests made at intervals of a few days.

It has not been possible to find just the reason for this behavior. The thermocouples and flow meters have been carefully and frequently checked, the time intervals always kept within a few

Table I

Effect of Small Quantities of Moisture in Air On the Scaling of Steel at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% of Moisture	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average
296	40	60	0	4.74	4.78	0.228	0.229		.320
337	40	50	0	4.82		0.230		.320	
411	40	60	1	5.10	5.14	0.244	0.246	.336	.340
412	40	60	1	5.19		0.249		.345	
409	40	60	3	5.54	5.63	0.267	0.270		.376
410	40	60	3	5.72		0.274		.376	
408	40	60	5	5.75	5.85	0.275	0.280		.405
413	40	60	5	5.96		0.285		.405	

seconds, and every condition that may vary has been subjected to rigid scrutiny. It has been possible, in general, to obtain results that check within three per cent of the average on tests run under the same conditions at intervals of a few days or a few months, and closer on tests run consecutively.

Since moisture in the air was found to interfere with the accuracy of results obtained, it was decided to find the effect of small quantities of moisture in air. Accordingly, tests were made with dry air, and with air containing one, three, and five per cent of moisture. These tests were made at 2300 degrees Fahr., with the gas flowing at the rate of 60 feet per minute and the steel samples placed in the furnace for a total time of 40 minutes. The correct percentage of moisture in the air was obtained by passing dried air through several bottles of water held at the correct temperature to give the required percentage of moisture when the air was saturated at that temperature. It was found necessary to prevent entrainment of water by means of a suitable trap, also maintained at the same temperature. Table I shows the results obtained and the variations that may be expected with moisture contents that are encountered in atmospheric air. Air saturated with moisture at 90 degrees Fahr., a condition often met in a forge shop, will contain five per cent of moisture.

Having determined the effect of moisture in the air on the scaling of steel, it was, of course, necessary to make all of the subsequent tests with dry air. The next series of tests made were for the purpose of determining the effect of the rate of flow of air on scaling at 2300 degrees Fahr. The rates of flow were increased in small increments from nearly 0 to 70 feet per minute. From 70 feet per minute the rate was increased to 120, then to 240, then to 385, and finally to 962 feet per minute. The duration of the tests in all cases was 40 minutes. All rates of flow are calculated on the basis of the tube cross-section, so that when flowing past the sample, the velocity will be increased about 20 per cent due to the space taken up by the sample.

The results of these tests presented in Table II show the surprising fact that with rates of flow higher than two feet per minute, that is, to 962 feet per minute, the same scaling loss is produced. Rates of flow higher than 962 feet per minute have not been tested. Decreasing the rate of flow to four feet per minute causes no change in the amount of scale produced and at two feet per minute the loss drops off slightly. At 0 rate of flow the scaling loss is less than 1/3 of that at two feet per minute. It is, of course, quite probable that at what is called 0 rate of flow there was some movement of the gas since it will be evident that it was impossible to prevent some movement of the gas in a two-inch tube of the length used in these tests. By 0 rate of flow is merely meant that both ends of the tube were sealed during the entire period of the test.

The weight of scale removed from the piece was determined as already described. This information serves as an independent check on the results of loss in weight provided of course that the average composition of the scale remains constant. The ratio of the loss in weight per 100 square inches to the weight of the scale per 100 square inches gives the average iron content of the scale. It will be observed that this ratio varies somewhat. This variation has been found to be greater when air or oxygen is the scaling medium than when carbon dioxide or steam is the scaling medium. The reason for this difference is not known. In plotting the results the loss in weight per 100 square inches is used in all cases rather than the weight of the scale.

The results are shown graphically in Figs. 2 and 3, Fig. 3 showing that portion of the curves under fifteen feet per minute presented

Table II

Effect of Variation of Rate of Flow of Dry Air on Scaling of Steel at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
340	40	nearly 0	1.31		.0628		.0836		
				1.33		.0638		.0847	0.752
347	40	nearly 0	1.36		.0648		.0857		
365	40	.5	2.10		.100				
				1.94		.092		.114	0.745
390	40	.5	1.77		.085		.114		
366	40	1.0	2.64		.126		.171		
				2.56		.122		.164	0.743
389	40	1.0	2.48		.118		.157		
367	40	1.5	3.31		.158		.219		
				3.25		.154		.213	0.722
388	40	1.5	3.18		.150		.208		
349	40	2.0	4.31		.206				
				4.36		.208		.293	0.716
353	40	2.0	4.41		.210		.293		
358	40	4.0	5.23		.252		.348		
				5.23		.252		.349	0.721
360	40	4.0	5.23		.253		.350		
351	40	6.0	5.07		.244		.340		
				5.08		.244		.341	0.715
359	40	6.0	5.09		.244		.342		
352	40	8.0	4.97		.242				
				4.95		.239			
356	40	8.0	4.94		.236				
333	40	10.0	4.84		.232		.317		
				4.90		.235		.317	0.730
341	40	10.0	4.97		.238				
334	40	20.0	4.85		.232				
				4.98		.239		.348	0.707
342	40	20.0	5.11		.246		.348		
335	40	30.0	4.60		.218		.300		
				4.69		.224		.300	0.726
343	40	30.0	4.77		.230				
336	40	40.0	4.74	4.74	.226	.226	.323	.323	0.70
337	40	50.0	4.82	4.82	.230	.230	.320	.320	0.718
296	40	60.0	4.74	4.74	.228	.228			
338	40	70.0	4.86	4.86	.233	.233			
339	40	120.0	4.83		.232		.328		
				4.82		.232		.328	0.707
344	40	120.0	4.81		.232				
345	40	240.0	4.79	4.79	.228	.228			
346	40	385.0	4.70	4.70	.226	.226			
559	40	962.0	4.80	4.80	.230	.230			

in Fig. 2. So far as is known, this effect of rate of flow has never been reported and is, of course, of very considerable importance. We believe it explains some of the wide variations in scaling results which have been reported in the literature.

A series of tests was next made to determine the effect of varia-

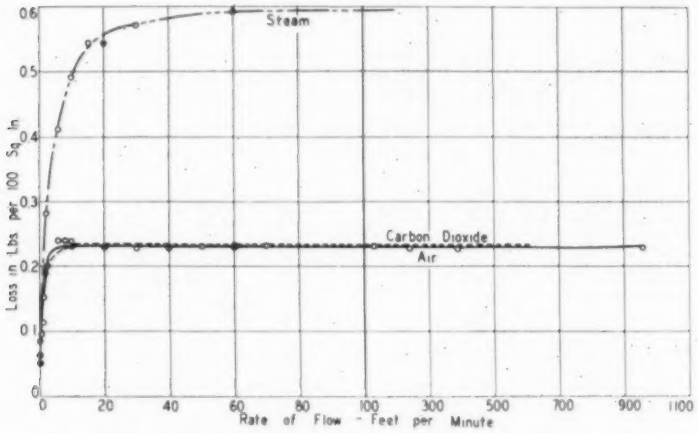


Fig. 2—Curves Showing Effect of Rate of Flow of Oxidizing Gases on Scaling of Steel.

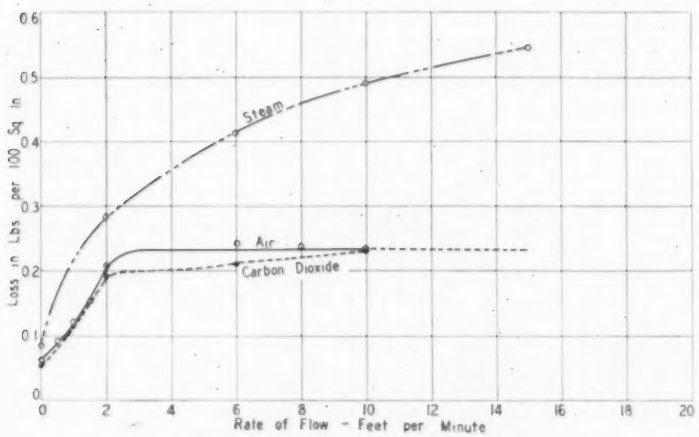


Fig. 3—Curves Showing Effect of Variation of Rate of Flow of Oxidizing Gases on Scaling of Steel.

tion in temperature on scaling. A uniform rate of flow of 60 feet per minute was used and each test was run 40 minutes. The effect of scaling was investigated at the temperatures of 2000, 2100, 2200, 2300, 2400, and 2475 degrees Fahr. The data obtained in these tests are summarized in Table III. Although it was expected that there would be an increase in the amount of scale produced with an increase in temperature, it was scarcely thought that such large differences would be found with relatively small changes in temperature. The results have been plotted and are displayed in Fig. 4. This curve shows rather regular increase in scaling with increase in temperature, in fact between 2000 and 2300 degrees Fahr. the curve is a straight line. There is a tendency to a greater increase in

Table III

Effect of Variation of Temperature on Scaling in Dry Air

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	Temperature Degrees Fahr.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
213	40	60	2000	2.66		.127		.175		
214	40	60	2000	2.68		.127		.180		
					2.58		.123		.172	0.716
293	40	60	2000	2.41		.115		.159		
317	40	60	2000	2.75		.124		.172		
216	40	60	2100	3.36		.162		.226		
294	40	60	2100	3.48	3.38	1.66	.162	.234	.231	0.702
318	40	60	2100	3.28		.159		.234		
295	40	60	2200	4.11		.197		.273		
					3.99		.190		.264	0.719
319	40	60	2200	3.87		.184		.255		
296	40	60	2300	4.74		.228				
337	40	50	2300	4.82	4.81	.230	.230	.320	.320	0.718
338	40	70	2300	4.86		.233				
268	40	60	2400	6.23		.298		.414		
					6.37		.305		.419	0.727
269	40	60	2400	6.50		.312		.424		
551	20	60	2475	24.4		1.175				

scaling with temperatures above 2300 degrees Fahr., and especially above 2400 degrees Fahr. In the tests made at temperatures of 2400 degrees Fahr. and above, the steel sample becomes hotter than the furnace atmosphere due to the exothermic reaction $3Fe + 2O_2 \rightarrow Fe_3O_4 + 270800$ calories. At these temperatures the rate of oxidation is rapid enough to increase the temperature in the heating chamber.

The results obtained with air at these temperatures, that is at 2400 degrees Fahr. and above, do not represent the amount of scale formed at the temperature since the steel becomes hotter than the furnace temperature. The results presented, therefore, merely represent the amount of scale formed when clean steel is placed in a furnace at these temperatures. In the case of Test No. 551, although the sample was placed in the furnace at 2475 degrees Fahr., the sample became so hot as to heat the surrounding atmosphere to 2540 degrees Fahr. This same phenomenon was observed in the previous work of one of the authors in connection with a study of the burning of steel.

For the tests made above 2400 degrees Fahr., a special refractory boat was used to support the sample. This boat was made from

chrompatch cement the shape of a segment of a cylinder about one inch longer than the steel sample, about 1½ inches wide and about ¼ inch thick to fit into the muffle tube. Two cross bars consisting of small segments of thermocouple insulator tubes were cemented on to the boat and the steel sample placed across these bars. By this means the tube was protected from iron oxide that might drip off the steel sample and the steel sample only touched the refractory in

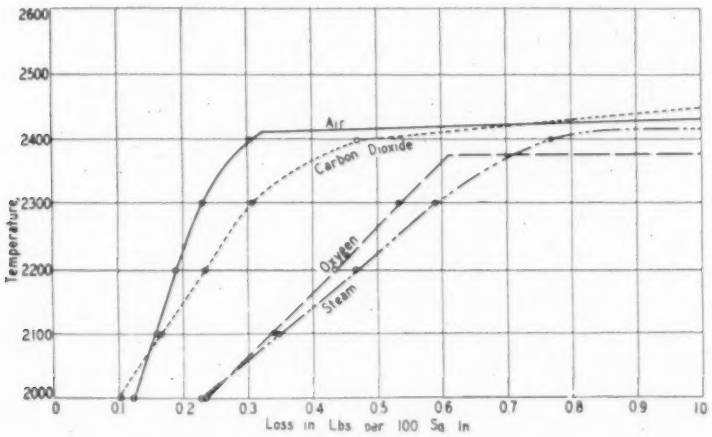


Fig. 4—Curves Showing Effect of Variation of Temperature on Scaling of Steel.

two spots. When this refractory boat was used, tongs were employed in placing the sample in the furnace and removing it. Since there was some fusion of the oxide and consequent dripping off of the sample, no attempt was made to determine the quantity of scale formed and the loss in weight was the only determination made.

To study the effect of variations in time of exposure on scaling, tests were made at 2300 degrees Fahr., in which the steel was exposed to the action of dry air for 5, 10, 15, 20, 40, 80, and 160 minutes. The rate of flow in all these tests was kept constant at 60 feet per minute. It must be borne in mind that the periods of exposure were for the total time in the furnace and not for the total time the sample was at heat. For instance, the samples heated for five minutes had barely reached 2300 degrees Fahr. when they were removed and quenched and consequently the amount of scale formed will represent what is formed while the sample is coming to heat. The results obtained are summarized in Table IV. Examination of this table will show, as might be expected, that greater time of exposure produced larger quantities of scale. However, as the time

Table IV

Effect of Variation of Time of Exposure on Scaling of Steel in Dry Air at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale lbs. per sq. in.	Average	Ave. Loss in Wt. / Av. Wt. of Scale
455	5	60	1.08	1.08	.0519	.0518	.0735	.0721	0.718
458	5	60	1.08		.0518		.0707		
454	10	60	2.33	2.39	.112	.115	.156	.161	0.714
459	10	60	2.45		.117		.166		
457	15	60	2.97	2.89	.140	.138		.192	0.703
461	15	60	2.82		.135		.192		
285	20	60	3.49	3.45	.168	.165	.233	.228	0.723
286	20	60	3.41		.162		.223		
287	40	60	4.69	4.67	.222	.222	.298	3.00	0.740
288	40	60	4.65		.221		.302		
289	80	60	6.60	6.50	.316	.311			
290	80	60	6.39		.305				
291	160	60	9.32	9.21	.447	.442			
292	160	60	9.10		.436				

of exposure is increased, the rate of scaling is progressively slower. This is shown graphically in Fig. 5. The equation of this curve has been found to be $W^2 = 0.001254t$ where W is the loss in weight in pounds per 100 square inches and t is the time of exposure in minutes. This equation will not hold so well for the points below 10 minutes for the reason just explained.

Oxygen

Only one series of tests has been made with oxygen. This series was made to study the effect of variation of temperature from 2000 to 2375 degrees Fahr. The oxygen used was the commercial tank oxygen which was found by analysis to contain 99.5 per cent oxygen. It will be observed form Table V that the loss in weight from scaling increases very rapidly with increasing temperature. It will be noted that at 2375 degrees Fahr. all the steel is converted to oxide, whereas at 2300 degrees Fahr. only 11.17 per cent of the iron is converted to oxide. This very decided increase in the rate of scal-

Table V

Effect of Variation of Temperature on the Scaling of Steel in Oxygen

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	Temperature Degrees Fahr.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
541	40	60	2000	4.85		.232				
					4.85		.232		.335	0.707
542	40	60	2000	4.85		.233		.330		
532	40	60	2100	7.16		.343		.465		
					7.22		.346		.480	0.721
533	40	60	2100	7.28		.349		.495		
530	40	60	2200	8.86		.424		.575		
					9.14		.438		.610	0.717
531	40	60	2200	9.42		.452		.646		
538	40	60	2300	10.75		.514		.705		
					11.17		.534		.742	0.720
539	40	60	2300	11.59		.554		.780		
					100					
545	10	60	*2375	100						

*Due to burning of sample, temperature became very much hotter than 2375 degrees Fahr. Although it was impossible to measure temperature of piece, it was hot enough to vaporize the iron oxide.

ing appears to be due to the higher temperature to which the steel becomes heated by reason of rapid reaction with oxygen. After the steel has been placed in the furnace, its temperature soon becomes so hot as to volatilize the iron oxide that is formed. The temperature registered by the thermocouple was above 2550 degrees Fahr. It increased so rapidly that it was scarcely possible to keep pace with the potentiometer reading. Since the thermocouple was separated from the piece by a refractory protection tube, it is impossible to know the actual temperature of the steel sample. There is evidence of the sample becoming hotter than the furnace atmosphere at 2300 degrees Fahr., but the temperature is not sufficiently high to initiate the reaction observed at 2375 degrees Fahr. At 2350 degrees Fahr. the heat generated was sufficient to cause the temperature in the tube to rise above 2400 degrees Fahr., but apparently the scale formed before the sample reached the temperature of very rapid reaction acted as a protecting agent. However, when a clean sample was placed in the tube at 2375 degrees Fahr., the combination with oxygen was so rapid that the sample became white hot and then burned completely to iron oxide. The iron oxide was volatilized and appeared as a dense smoke. It diffused through to

the inside of the thermocouple refractory protection tube as was shown by the deposit of iron oxide on the inside of the tube. The thermocouple wires were contaminated and had to be cut back about two inches from the end and rewelded. This contamination of thermocouple wires was similar to that reported by one of the authors[8] in connection with a study of the burning of steel.

Steam

The action of steam on scaling was investigated to determine the effect of rate of flow of the steam, the effect of variations in temperature, the effect of time of exposure, and the effect of admixture with nitrogen.

The steam was prepared from distilled water freshly boiled to eliminate dissolved gases. A small boiler was constructed by means of a 4-inch pipe, three feet long, capped at both ends and set vertically. An electric immersion heater was placed in the lower end of the tube by screwing it into the lower cap which had been previously tapped for this purpose. In the upper cap were placed three valves, one for introducing water, one for bleeding off the steam, which was used only before the test was commenced, and a third for conducting the steam to the furnace. The steel pipe was insulated with an inch of asbestos. In filling the boiler an air space of at least 10 inches between the surface of the water and top of the boiler was always maintained. By means of a wattmeter and a condenser, it was possible to calibrate the boiler so that a measurement could be made of the rate of flow of the steam from the number of watts used to heat the immersion heater. The steam line running from the boiler to the furnace was heated by means of electric current passing through a coil of wire wound around the steam line. This equipment was used for all the steam tests except in the tests with various mixtures of nitrogen and also where the rate of flow was 20 feet per minute or less. Since for these lower rates of flow the boiler could not be controlled with sufficient accuracy, a smaller boiler was constructed for this purpose. This smaller boiler was made from a glass tube the shape of a test tube having a capacity of 1500 cubic centimeters of water. An electric immersion heater was placed in the top of this tube. Water

[8]W. E. Jominy, TRANSACTIONS, American Society for Steel Treating, Vol. 16, 1929, p. 298.

Table VI
Effect of Variation of Rate of Flow on Scaling of Steel in Steam at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
486	40	0	1.76	1.78	.0845	.0855	.1100	.1116	0.766
487	40	0	1.80		.0865		.1133		
478	40	2	5.80	5.73	.2940	.2830	.383	.367	0.770
479	40	2	5.67		.2720		.352		
524	40	6	8.70	8.60	.415	.411	.531	.530	0.774
525	40	6	8.49		.407		.530		
522	40	10	10.31	10.22	.4970	.491	.647	.641	0.766
523	40	10	10.12		.4850		.635		
526	40	15	11.50	11.40	.553	.546	.723	.716	0.763
527	40	15	11.29		.540		.710		
528	40	20	11.45	11.36	.550	.546	.720	.714	0.765
529	40	20	11.28		.542		.709		
496	40	30	11.94	11.96	.571	.572	.742	.748	0.765
497	40	30	11.99		.573		.754		
498	40	60	12.49	12.39	.596	.592	.779	.774	0.765
499	40	60	12.29		.588		.770		
534	20	60	7.50		.359	.359	.468	.468	0.767
*113	20	172	7.56		.351				

*Test made in a steel tube instead of porcelain tube.

was introduced into the tube through a small glass tube sealed into the bottom of the boiler and by means of a large container at a suitable height the water was kept at a constant level in the boiler tube. With this smaller boiler, rates of flow in the range of 2 to 20 feet per minute could be maintained within an accuracy of 0.1 feet per minute flow in the furnace tube.

The results of the tests on the effect of variation of the rate of flow are shown in Table VI. It will be observed that the amount of scale produced at two feet per minute is more than 3 times the amount produced when the steam is still.

Further, at a rate of flow of 15 feet per minute, the amount of scale produced is more than double that at two feet per minute. Varying the rate of flow from 15 feet to 30 feet per minute appears to cause a very small increase in the amount of scale and from 30 to 60 feet there appears to be a slight increase. Increasing the rate of

Table VII

Effect of Variation of Temperature on the Scaling of Steel in Steam

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	Temperature Degrees Fahr.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
235	40	60	2000	4.70		.224		.286		
					4.78		.228		.297	0.768
236	40	60	2000	4.87		.232		.292		
237	40	60	2100	7.06		.338		.441		
					7.12		.342		.446	0.767
238	40	60	2100	7.19		.347		.451		
239	40	60	2200	9.74		.470		.616		
					9.72		.467		.610	0.765
240	40	60	2200	9.70		.464		.605		
498	40	60	2300	12.49		.596		.780		
					12.39		.592		.775	0.762
499	40	60	2300	12.29		.588		.771		
266	40	60	2400	16.38		.781		1.030		
					16.10		.770		1.012	0.760
267	40	60	2400	15.83		.760		.995		
547	20	60	2500	30.4	30.4	1.452				

flow from 60 to 172 feet per minute causes no increase in the amount of scaling. The only test made at 172 feet per minute was made in a steel tube during the early work in this study. It is believed that at this high rate of flow the effect of the steel tube is practically negligible.

It will be observed from Table VII that increase in temperature causes a large increase in the rate of scaling. At 2400 degrees Fahr., more than three times as much scale is formed in the same period of time as is formed at 2000 degrees Fahr. When the temperature is increased to 2500 degrees Fahr., the rate of scaling is very rapid, so rapid, in fact, that the hydrogen generated by the reaction burns with a steady flame at the crevices between the front plug and the tube. So much scale was formed in a period of 20 minutes that the run was discontinued at this time because it was feared that the tube would be burned out from the action of the iron oxide. For the 2500 degree Fahr test, the sample was placed on a specially constructed boat made of "chrompatch" with sillimanite cross bars and so supported that it touched the cross bars at only two points in the same manner as that described for the high temperature air tests. After the 20-minute test, the chrompatch boat was found to be fused and disintegrated. The porcelain tube, however, remained in

Table VIII
Effect of Variation of Time of Exposure on Scaling of Steel in Steam at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
534	20	60	7.50	7.48	.359	.357	.468	.468	0.767
535	20	60	7.45		.355		.467		
498	40	60	12.49	12.39	.596	.592	.779	.774	0.766
499	40	60	12.29		.588		.770		
488	80	60	20.55	20.67	.981	.991	1.281	1.295	0.765
489	80	60	20.80		1.000		1.309		
490	160	60	31.00	31.10	1.481	1.488	1.956	1.947	0.766
491	160	60	31.20		1.495		1.938		

fair condition. It was found that the oxide had fused and run off the steel, so that it presented clean steel continuously to the action of the steam. It is believed that the fusion of the scale permitting a fresh surface to be continuously brought in contact with the steam was the prime cause for the very marked increase in the rate of scaling at 2500 degrees Fahr.

The effect of variation of time of exposure on scaling in steam at 2300 degrees Fahr. is shown in Table VIII. This shows an increase in the amount of scale produced with increasing time, as was expected. The rate of scaling, however, decreases with increase in time of exposure. This is shown graphically in Fig. 5. The equation for this curve has been found to be $W^2 = 0.01552t - 0.2695$ where W is the loss in weight per 100 square inches and t is the time of exposure in minutes.

Mixtures of nitrogen and steam were passed over steel in order to study the effect of the action of steam under reduced pressure. Since nitrogen is known not to scale steel, diluting steam with nitrogen would, of course, have the effect of reducing the pressure of steam. The nitrogen used was the commercial tank nitrogen. For producing the mixtures from 1 to 10 per cent, the nitrogen was passed through the humidifier already described in connection with the air-moisture tests. For mixtures from 15 to 50 per cent steam, the steam from the boiler was mixed with the nitrogen. It

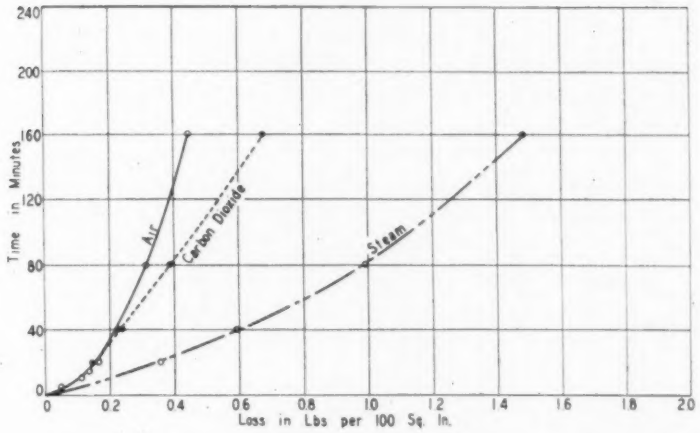


Fig. 5—Curves Showing Effect of Period of Exposure on Scaling of Steel at 2300 Degrees Fahr.

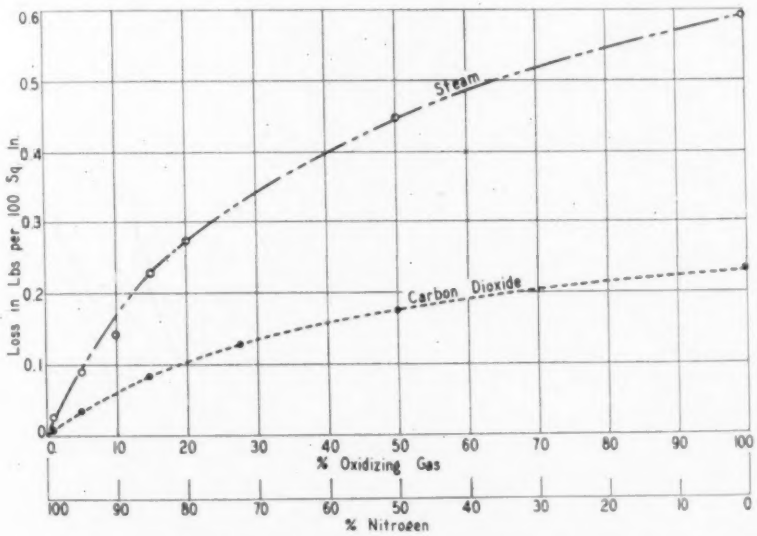


Fig. 6—Curves Showing Effect of Dilution of Oxidizing Gases with Nitrogen on Scaling of Steel.

may be seen from Table IX and Fig. 6 that decreasing the vapor pressure of the steam decreases the rate of scaling of the steel.

Carbon Dioxide

The carbon dioxide used in these tests consisted of the commercial tank carbon dioxide which was found to vary in composition from 99.85 to 99.95 per cent carbon dioxide. Tests were made to determine the effect of rate of flow of this gas on scaling at 2300 degrees Fahr., the effect of variation in temperature, the effect of time of exposure, and the effect of decreasing the pressure.

Table IX

Effect of Mixtures of Steam and Nitrogen on the Scaling of Steel at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% N_2	% H_2O	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
403	40	60	99.0	1.0	0.65	0.56	.0310	.0268			
404	40	60	99.0	1.0	0.47		.0226		.0301	.0301	0.751
405	40	60	95.0	5.0	1.90	1.88	.091	.090	.1144	.1154	0.778
407	40	60	95.0	5.0	1.86		.089		.1164		
414	40	60	90.0	10.0	2.92	3.02	.140	.1449	.1834	.1883	0.767
415	40	60	90.0	10.0	3.13		.1498		.1936		
392	40	60	85.0	15.0	4.68	4.82	.224	.230		.3022	0.780
397	40	60	85.0	15.0	4.95		.236		.3022		
393	40	60	80.0	20.0	5.78	5.72	.276	.273	.3573	.3573	0.774
398	40	60	80.0	20.0	5.66		.271				
395	40	60	50.0	50.0	9.46	9.35	.453	.448	.592	.785	0.765
400	40	60	50.0	50.0	9.25		.443		.579		
498	40	60	0.0	100.0	12.49	12.39	.596	.592	.779	.774	0.765
499	40	60	0.0	100.0	12.29		.588		.770		

The effect of variation of rate of flow of carbon dioxide at 2300 degrees Fahr. seems to parallel the effect produced with air and steam. As may be observed from Table X the amount of scale produced at zero rate of flow is less than one-third that produced when the rate of flow was two feet per minute. Increasing the rate of flow from two to 10 feet per minute causes a small increase in the amount of scale. From 10 feet to 570 feet per minute, no change in the amount of scaling is obtained. Tests 93 and 100 were run during the preliminary work on this study and were consequently tested in a steel tube. In the case of these high rates of flow, the effect of the steel tube in changing the gas composition is negligible. Periods of exposure of 20 minutes were used in these latter tests to conserve the carbon dioxide.

Table XI shows the effect of variation of temperatures from 2000 to 2500 degrees Fahr. It will be observed that the rate of scaling is more than four times as rapid at 2400 degrees Fahr. as that at 2000 degrees Fahr. The tests at 2500 degrees Fahr. show a very decided increase in the amount of scale produced. These

Table X
Effect of Variation in Rate of Flow of Carbon Dioxide on Scaling of Steel at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
442	40	0	1.17	1.15	.0559	.0549			
444	40	0	1.13		.0540		.0696	.0696	0.775
443	40	2	3.98	4.02	.1905	.1927	.252	.252	0.763
445	40	2	4.06		.1950		.253		
446	40	6	4.39	4.36	.210	.209	.272	.270	0.772
473	40	6	4.33		.208		.269		
447	40	10	4.78	4.80	.2285	.2302	.298	.301	0.766
449	40	10	4.82		.2320		.304		
450	40	20	4.85	4.71	.232	.228	.300	.297	0.767
452	40	20	4.67		.224		.294		
451	40	60	4.85	4.81	.232	.230	.303	.298	0.770
453	40	60	4.77		.228		.294		
536	20	60	3.00		.144		.188		0.765
* 93	20	285	2.94		.140		.188		0.745
*100	20	570	3.14		.149		.203		0.732

*Test run in a steel tube.

latter tests were made with the refractory boat as already described. Only 20-minute tests were made in order to favor the porcelain tube. The scale had not melted and run off the sample as occurred when the steel was exposed to steam at 2500 degrees Fahr., although the surface of the scale appeared glazed. The refractory boat appeared to be as satisfactory after the test as before, also indicating that very little, if any, iron oxide had come in contact with the boat. The rate of scaling was sufficiently rapid to cause so much carbon monoxide to be formed that it burned in the crevices between the front plug and the tube.

In Table XII are presented the data showing the effect of variation of time of exposure to carbon dioxide at 2300 degrees Fahr. These results show the same general tendency as are shown by air and steam, that is, the amount of scale increases with time of exposure, but the rate of scaling decreases with time of exposure.

The results obtained when carbon dioxide was diluted with nitrogen and passed over steel at 2300 degrees Fahr. are shown in

Table XI
Effect of Temperature Variation on the Scaling of Steel in Carbon Dioxide

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	Temperature Degrees Fahr.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
271	40	60	2000	1.62	1.62	.0775	.0777	.102	.102	0.762
272	40	60	2000	1.63		.0780		.102		
273	40	60	2100	2.58	2.67	.124	.128	.162	.165	0.772
274	40	60	2100	2.77		.132		.167		
275	40	60	2200	3.66	3.73	.1755	.1785	.228	.232	0.766
276	40	60	2200	3.80		.1815		.237		
277	40	60	2300	4.94	4.90	.237	.234	.308	.305	0.766
451	40	60	2300	4.85		.232		.303		
520	40	60	2400	7.45	7.43	.357	.355	.467	.464	0.766
521	40	60	2400	7.41		.354		.461		
548	20	60	2500	16.62	16.51	.798	.792			
549	20	60	2500	16.41		.786				

Table XIII. Since nitrogen is inert, these data may be considered to show the effect of reducing the pressure of the carbon dioxide. A general decrease in the rate of scaling is observed as the pressure is decreased. Steam was observed to exhibit this same tendency.

Comparison of the Action of the Gases Tested

A comparison of the scaling effect of the four gases, air, oxygen, steam, and carbon dioxide, may be made by reference to Fig. 4. The points for carbon dioxide and steam at 2500 and air at 2475 degrees Fahr. were obtained by 20-minute exposures instead of the 40-minute exposures used for the rest of the tests presented in this figure. The curves were therefore not drawn to these points and the slope of the curve is for this reason estimated beyond 2400 degrees Fahr. It will be observed that the air curve and carbon dioxide curve cross in two points, just a little below 2300 degrees Fahr. and at 2425 degrees Fahr. The crossing at 2425 degrees Fahr. is, the authors believe, due to the exothermic reaction between air and steel generating so much heat that the sample becomes hotter than the furnace atmosphere. No such reaction occurs with carbon dioxide so that its curve follows the normal course. It is of

interest to note that between the temperature of 2000 and 2300 degrees Fahr., each one of the curves is a straight line.

It has been rather surprising to the authors to find steam such an active oxidizer at these temperatures. Reference to Fig. 4 shows that practically the same amount of scale is produced with pure steam as with pure oxygen between the temperatures 2000 and 2300 degrees Fahr. However, at 2375 degrees Fahr., oxygen shows its superiority to steam by causing 100 per cent conversion of iron to iron oxide in 10 minutes time, whereas steam at 2400 degrees Fahr. causes 16.1 per cent conversion in 40 minutes time. This sudden increase in the rate of oxidation caused by pure oxygen is primarily due to the exothermic reaction already referred to. The heat generated when steel is placed in the furnace at this temperature is sufficient to cause a fusion of the oxide and then, because of the continued rapid oxidation of the thus unprotected steel, causes the melting of the steel itself. If the steel is allowed to become coated with solid oxide at temperatures such as 2325 or 2350 degrees Fahr., it can then be heated to 2400 degrees Fahr. without initiating the fast rate of oxidation that causes fusion of the sample. This shows the protective action of the oxide scale. This protecting action of the oxide formed is also evidenced by the decreasing rate of oxidation with increased time of exposure, as may be observed in Table IV, VIII, and XII and in Fig. 5. The protecting action of the scale formed on the steel is of considerable importance. Its physical and chemical composition will determine, in part, the rate of oxidation of the steel. The physical characteristic of the scale chiefly affecting the rate of scaling is, we believe, its permeability to the scaling gases.

According to Pfeil,[9] oxidation of steel in air produces at least three layers of oxide, in which the outside layer contains the highest percentage of oxygen, the middle layer less oxygen, and the inside layer contains the least oxygen. This observation has been made by other investigators and there appears little reason to doubt the soundness of the conclusion. Whether these oxides are composed of various mixtures of FeO and Fe_3O_4, as indicated by Pfeil, or are produced by mixing with iron itself, is still an open question.

There is a distinct difference in the appearance of the oxides formed by the different gases. The scale formed with steam has a well developed structure having a coarsely crystalline, shiny ap-

[9] L. B. Pfeil, *Journal*, Iron and Steel Institute, Vol. 119, 1929, p. 501.

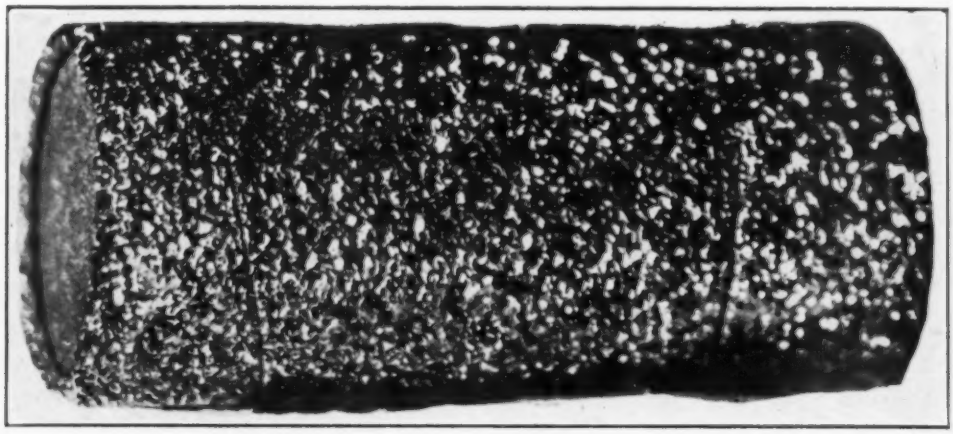


Fig. 7—Scale on Surface of Sample Subjected to the Action of Steam at 2300 Degrees Fahr. Magnification × 2.

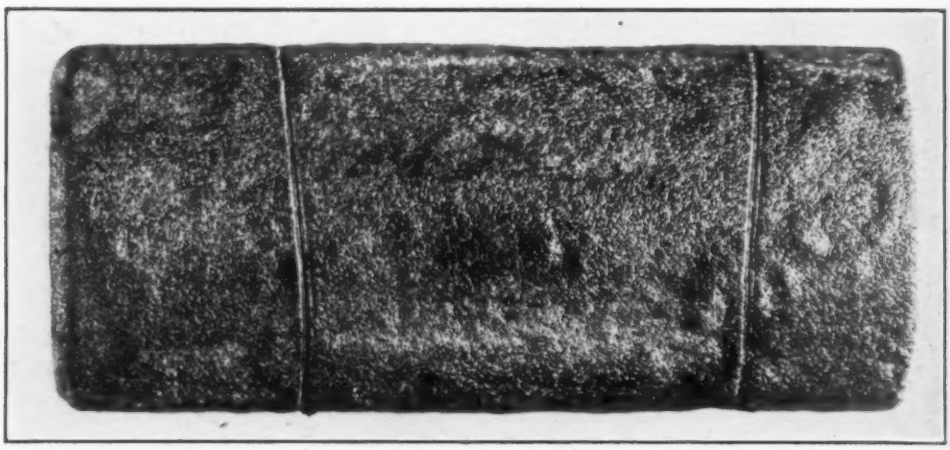


Fig. 8—Scale Formed on Surface of Sample Subjected to the Action of Dry Air at 2300 Degrees Fahr. Magnification × 2.

pearance. The surface that is formed at 2300 degrees Fahr. is very rough, and when quenching, the scale peels off the steel in relatively large pieces. The scale formed with carbon dioxide resembles in crystalline form that produced with steam. It is not quite so coarse and upon quenching splits off the steel in rather finer flakes. It would appear to have a higher melting point since it does not fuse off the steel at 2500 degrees Fahr., whereas the scale formed with steam does fuse at this temperature. The scale produced in air or in oxygen has a smooth glossy appearance and shows no grain structure. The appearance of the scale produced with steam is shown in Fig. 7 and that produced in air is shown in Fig. 8.

In addition to the difference in physical appearance there seems

Table XII

Effect of Variation of Time on Scaling of Steel in Carbon Dioxide at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
536	20	60	3.00	3.11	.143	.149	.1866	.1898	0.768
537	20	60	3.23		.155		.203		
277	40	60	4.94	4.90	.237	.234	.308	.305	0.766
451	40	60	4.85		.232		.303		
474	80	60	8.04	8.07	.386	.387	.498	.500	0.773
475	80	60	8.10		.388		.502		
476	100	60	14.70	14.14	.701	.675	.909	.877	0.769
477	100	60	13.59		.650		.846		

to be a distinct difference in chemical composition of the scale formed in the different atmospheres. When the average loss in weight per 100 square inches is divided by the average weight of scale per 100 square inches, the result should approximate the average iron content of the scale. These ratios are presented in Tables II to XIII. For some reason unknown to the authors these ratios when carbon dioxide or steam is used are much more constant than when air or oxygen is the scaling medium. For air, it will be seen that the iron content is reasonably close to that of Fe_3O_4; but for steam and carbon dioxide, the ratios indicate a composition very near FeO. In order to check further the iron content indicated by these ratios, chemical analyses for total iron were made on a few typical samples of scale produced with the different atmospheres.

Samples 337 and 339, which were scaled in air, were found by analysis to contain 72.3 and 72.4 per cent of iron respectively. It will be noted from Table II that the ratio of loss in weight over weight of scale for Sample 337 was 71.8 per cent and for 339 was 70.7 per cent. The percentage of iron in Fe_3O_4 is 72.4 per cent.

Samples 486, 487, 497 and 499, which were produced by the action of steam, were analyzed for iron content. Below are listed the per cent of iron obtained by chemical analysis of the scale and the loss in weight over weight of scale ratios of these samples:

Sample No.	% Fe in Scale by Analysis	Ratio of loss in weight to weight of scale from Table VI as per cent
486	76.2	76.8
487	76.2	76.6
497	76.4	76.2
499	76.0	76.4

In all of these samples there is fairly close agreement between the percentage of iron in the scale as determined by chemical analysis and that determined from the ratio of loss in weight to weight of scale. The percentage of iron in FeO is 77.6 per cent.

Samples 450 and 453, which were oxidized in carbon dioxide, were found to contain 76.1 and 76.0 per cent respectively. These values may be compared with the ratios given for these samples in Table X which for sample 450 is 77.3 per cent and for sample 453 is 77.4 per cent.

It is evident from the agreement of the ratios in all of the data presented in Tables II to XIII and from these chemical analyses of typical scales that the average iron content of scale found in air approaches that of Fe_3O_4 and the average iron content of scale found in carbon dioxide or steam approaches that of FeO. Apparently the chemical as well as physical characteristics of the scale depend on the atmosphere employed.

The mechanism of oxidation with air, as postulated by Pfeil, seems to be in agreement with the results obtained in this investigation for air. Pfeil's mechanism of oxidation consists of the inward diffusion of oxygen and the outward diffusion of iron through the scale. It is at once evident that as the thickness of the scale increases, the rate of oxidation will decrease, due to the necessity of both the oxygen and iron diffusing through thicker layers.

The effect of rate of flow of the air seems to be at least in part due to the diffusion rate of the oxygen through the scale. When the oxygen is not supplied rapidly enough to replace that removed by diffusion through the scale, a certain reduced rate of oxidation occurs, determined by the rate at which the oxygen is supplied. This is in part a matter of the oxygen concentration of the gas immediately surrounding the sample. When the oxygen is supplied faster than it can be removed by the diffusion of oxygen through the scale no more rapid rate of oxidation can occur. In other words, rates of flow greater than that necessary to balance the rate of diffusion will cause no increase in the oxidation rate. This is precisely what has

Table XIII
Effect of Mixtures of Carbon Dioxide and Nitrogen on the Scaling of Steel at 2300 Degrees Fahr.

Sample Number	Total Time in Furnace, Min.	Rate of Flow Ft. per Min.	% N_2	% CO_2	% Loss in Weight	Average	Loss in Lbs. per 100 sq. in.	Average	Wt. of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Wt. / Av. Wt. of Scale
320	40	60	99.0	1.0	0.15		.0071		.0095		
						0.18		.0086		.0112	0.768
328	40	60	99.0	1.0	0.21		.0101		.0128		
321	40	60	94.9	5.1	0.64		.0305		.0396		
						0.75		.0362		.0468	0.773
329	40	60	94.9	5.1	0.87		.0420		.0540		
322	40	60	85.4	14.6	1.80		.0865		.111		
						1.78		.0852		.110	0.777
330	40	60	85.4	14.6	1.76		.0840		.108		
323	40	60	72.8	27.2	2.70		.1297		.169		
						2.73		.1309		.170	0.770
331	40	60	72.8	27.2	2.76		.1326		.171		
324	40	60	50.0	50.0	3.71		.177		.228		
						3.71		.177		.228	0.774
332	40	60	50.0	50.0	3.70		.177		.229		
277	40	60	0.0	100.0	4.94		.237		.308		
						4.89		.234		.305	0.766
451	40	60	0.0	100.0	4.85		.232		.303		

been observed in the tests. This rate would be expected to vary with variations in temperature and would probably increase with increase in temperature due to the probably greater speed of diffusion of the oxygen through the oxides at the higher temperature.

In the case of the scaling with steam, there is a question as to what occurs. The molecular water may diffuse through the scale and finally decompose into hydrogen and oxygen, after which the oxygen is fixed and the hydrogen must diffuse back out; or the molecule of water may be broken up at the outer surface of the scale forming atomic oxygen which could diffuse much more easily or possibly forming molecular oxygen which would diffuse more slowly. Certainly the appearance and looseness of structure of the scale formed with steam is very different from that formed with air or that formed with oxygen. The scale formed with air had an appearance similar to that of scale formed with oxygen. Whether the crystal structure or the composition of the scale differed remains to be determined.

Similarly, the mechanism of the reaction with carbon dioxide requires further investigation. Here it is a question whether the carbon dioxide gas diffuses through the scale to the surface of the

steel, or breaks up into carbon monoxide and oxygen at the surface of the scale after which the atomic, or perhaps molecular, oxygen diffuses through the scale toward the iron. The appearance of the scale resembles that produced by the steam, but a much smaller quantity is formed under similar conditions of temperature, time, and rate of flow.

The effect of variation in the rate of flow of the three gases, air, carbon dioxide, and steam, on scaling is quite similar, as may be observed from Fig. 2. In the case of all three gases, the most decided difference in rate of scaling is produced between zero and two feet per minute. With air there is only a slight increase in the rate of scaling produced at six feet per minute and no change in the rate of scaling by increase in the rate of flow beyond this value. With carbon dioxide the scaling rate is affected somewhat by increasing the rate up to ten feet per minute and increasing the rate of flow beyond this point does not affect the scaling rate. With steam, the change in rate of scaling is more gradual, increasing to thirty feet per minute. Increasing the rate of flow from 30 to 60 feet per minute causes a very slight increase in scaling, and beyond 60 feet per minute there is no additional increase in scaling rate. It is noteworthy in the case of each of the gases that beyond 60 feet per minute there is no effect from increasing the rate of flow. While this is only strictly true at 2300 degrees Fahr., it is expected that the same general behavior would be observed at other temperatures at which different values would be found for the point of minimum rate of flow beyond which no increase in rate of scaling is produced by increasing the rate of flow.

Comparing the effect of time of exposure at 2300 degrees Fahr., as shown in Fig. 5, it will be observed that although air and carbon dioxide produce very nearly the same quantity of scale after 20 and 40-minute exposures, the 80-minute exposures show a decided difference with the carbon dioxide producing the greater amount of scale. With 160-minute exposures this difference is definitely widened. It is probable that the scale formed with carbon dioxide permits easier diffusion or solution of the oxidizing gas than does the scale formed with air so that when the thickness of the scale becomes appreciable its effect becomes noticeable.

Mixing either steam or carbon dioxide with nitrogen reduces the amount of scale formed, as shown in Fig. 6. Since nitrogen produces no scale and has no effect, the dilution with nitrogen produces

the same effect of reduced pressure of the active gas and affords an easy means of finding the effect of reduced pressure.

Although with nitrogen mixtures a uniform curve of scaling loss is obtained, it would be unsafe to predict the scaling effect of a mixture of two gases which individually caused scaling. Due to the possibility of variation in the physical and chemical properties of the scale produced, such a prediction would be unwise. For instance, in the products of combustion of ordinary city gas burned with just the quantity of air to produce complete combustion, will be found nitrogen, carbon dioxide and steam. Such a mixture might contain about 17 per cent steam, 13 per cent carbon dioxide and 70 per cent nitrogen. According to the results obtained, it would be expected that with 17 per cent steam and 83 per cent nitrogen 5.20 per cent of iron would be removed by scaling and with 13 per cent carbon dioxide and 87 per cent nitrogen 1.60 per cent of iron would be removed. The reasoning thus far is correct, but it would be erroneous to assume that with the combination of these gases one should expect an amount of scaling equivalent to the sum of the two values or 6.80 per cent loss. Results of tests have shown that the loss in weight is much lower than may be expected with 17 per cent steam and 83 per cent nitrogen, even without the addition of the 13 per cent of carbon dioxide. A different type of scale is formed than with steam or carbon dioxide and it is believed that this scale acts as a better protector than that formed with the steam.

Summary

As a result of scaling tests made on plain carbon steel at forging temperatures air, steam, carbon dioxide and oxygen, the following generalizations seem justified:

1. Increasing the rate of flow of the gas between 0 and 2 feet per minute causes a very marked increase in the rate of scaling at 2300 degrees Fahr. With air or carbon dioxide, relatively little change in the rate of scaling is produced by increasing the rate of flow from 2 to 10 feet per minute. With these gases no change is produced with rates of flow higher than 10 feet per minute. In the case of steam, a noticeable increase in the rate of scaling occurs with increase in rate of flow from 2 to 15 feet per minute, and a very small increase in scaling rate up to 60 feet per minute, after which increasing the rate of flow has no effect on rate of scaling.

2. Increase in temperature causes increase in scaling rate. Between 2400 and 2500 degrees Fahr., the scaling rate increases sharply. When heated to 2375 degrees Fahr. in nearly pure oxygen, ordinary plain carbon steel oxidizes so rapidly that it is melted by the heat liberated in the chemical reaction with oxygen. When heated to about 2425 degrees Fahr. in air, the steel becomes hotter than the furnace atmosphere, but does not liberate enough heat to melt a low carbon steel.

3. Increase in time of exposure causes increase in the amount of scale produced, but decrease in the rate of oxidation.

4. Scale on steel acts as a partial protection against further scaling. The type of scale formed has a decided influence on the further rate of scaling.

5. Under the conditions obtaining in this research the average iron content of the scale formed with air approaches that of Fe_3O_4, and the average iron content of scale formed in carbon dioxide or steam approaches that of FeO.

Acknowledgments

The authors wish to express their appreciation to the American Gas Association whose financial support made this study possible and to William G. Frederick who made most of the chemical determinations.

DISCUSSION

Written Discussion: By A. M. Steever, metallurgical engineer, Great Lakes Forge Co., Chicago.

In reviewing Messrs. Jominy and Murphy's paper on the "Scaling of Steel at Forging Temperatures" it can be said that the authors recognize the importance of eliminating scaling which is very detrimental to the manufacture of forgings.

It is a well known fact that open flame furnaces cause oxidation of the steel or iron being heated to high temperatures producing much scale. This scale re-acts with most refractories and forms a flux of low melting point. This flux is very destructive and must be drained off the bottom of the furnace through a slag hole. This scale refractory adheres to the bars during the process of forging and causes no end of trouble, due to the fact that it is pounded into the surface of the part being made.

Slag can be eliminated by the use of automatic temperature control, and by this method reduce the working temperature to a point below the melting point of the scale produced. By eliminating the scale slag and producing dry scale we have a condition that is again very undesirable because of the fact that

it acts as an abrasive on the forging dies, shortening their life. It is pounded into the metal being worked, causing large scrap losses, due to the embedded scale affecting machinability and producing cavities upon the surface of the forging. A large loss of steel is also experienced by the excessive scaling in heating. There are many other destructive effects than can be attributed to the action of scale.

The authors have made some very interesting tests on both the production and elimination of scale at high temperatures. We, in the forging industry, appreciate this valuable research and have recognized the fact that, due to the destructive effects on our product, the decreased life of dies plus the large losses of steel caused by scaling, that eventually the scaling of steel at high temperatures must be reduced to a minimum.

In conclusion I wish to mention that we are experimenting in our plant with a furnace that will reduce scaling to a minimum. This is being accomplished by placing the bars being heated in an atmosphere of raw gas, keeping the excess oxygen away from the bars, preventing excessive scaling. The steel is heated by maintaining a luminous flame over the steel above this raw gas layer for the purpose of obtaining quick heat transmission.

Written Discussion: By Harold F. Wood, metallurgist, Ingalls-Shepherd Division, Wyman-Gordon Co., Harvey, Ill.

The authors of this paper are to be congratulated on the contribution that they have made to the knowledge of the factors affecting the scaling of steel at forging temperatures, and on the most thorough and painstaking way in which they carried out their researches.

The forging industry is most vitally interested in learning all that it can on this very important problem which they deal with on 100 per cent of their product. This question is of greater importance today than ever before. In the forging industry we are going through a very difficult period in which every item affecting costs is being studied from every possible angle in the hope that such study will develop ways and means of better control and lower costs. In the last few years there have been great improvements made in the method of heating steel for forging. This has consisted for the most part in the use of continuous furnaces under automatic temperature control wherever possible. The use of such equipment has resulted in more uniform heating because of its slower rate of heating and longer time in furnace. This in turn has resulted in reducing liability of burning and overheating to a minimum, thereby making it possible to secure consistently higher and more uniform properties in the finished parts. This slower rate of heating and longer soaking time in the high temperature zone has materially increased the amount of scaling. Part of this increase has been offset by forging at somewhat lower temperatures. The greatest difficulty arises on this type of equipment when delays occur that cannot be anticipated resulting in a large quantity of steel being left in the furnace. Very often such delay is so short that excessive scaling does not take place and a great many times the cause for delay cannot be corrected as soon as first thought and excessive scaling results. It is not uncommon to find it necessary to remove the steel from the high temperature zone of the furnace and then reheat same later. Even then the losses due to forgings not properly filling out are very great. Equally as serious is the upsetting of equilibrium conditions

in the furnace and the additional delay that occurs in re-establishing these conditions after cause of original delay has been corrected. Very often this additional delay is two or three times as long as the time required to correct cause of original delay. When one considers that such a delay is holding up production very often on several pieces of forging equipment which may carry a labor and overhead rate as high as $50.00 per hour, it can be easily seen that this is a most vital factor affecting costs. When equipment of this kind is only operated day turn, the question of overnight standover losses cannot be forgotten. Even though the furnaces are partly unloaded at night, it is necessary to push stock into the high temperature zone before forging operations begin in the morning. Seventy-five per cent of the forgings made that are not properly filled out are made during the first hour of operation in the morning. The majority of these are caused from excessively scaled steel. This excessive scaling retards production very greatly, even though there is enough stock to fill the forging due to the time consumed in blowing scale in order to produce a clean forging. Excessive scaling causes a great deal of scrap forgings not only through shy stock, but also from scale holes resulting from scale being driven into surface of forging during forging operation. This is especially true in the forging of alloy steels. The alloy scale seems to have a habit of sticking to the steel much harder than in the case of carbon steel. A hammer crew that has been forging carbon steel will produce five times as many forgings showing scale holes if forging alloy steel unless the rate of forging is greatly reduced, allowing more time for the blowing of scale from the forging and the die. The drop forger's troubles from scaling are not over by any means after the forging is made. Plenty of grief is encountered in the cleaning of the forgings before final inspection and shipment. A forging that has been water quenched is much easier to clean than one that has been annealed or normalized as the quenching operation removes the majority of the scale. For instance, it takes twice as long to pickle a normalized forging as it does to pickle a quenched and drawn forging and twice as long to pickle an alloy steel forging as a carbon steel forging. This means dollars and cents in a hurry.

This brief description of how scaling builds up costs in the forging industry shows the wonderful possibilities if these researches can be carried on to completion. It is hoped that it will be possible for this work to continue in the future. As a suggestion, it is hoped that future investigation will cover the several types of steels commonly used in the forging industry and that this be followed by an exhaustive research into ways and means of commercially controlling these factors so that scaling in industrial furnaces can be reduced to a minimum. I am very strongly of the opinion that some very great improvements can be worked out by a more careful control of furnace atmospheres. I am at the present time running experiments along this line. CO_2 recorder records from a previously thought perfect normalizing practice show curves that look like a kilowatt input curve on a stone crusher. Careful burner regulation will straighten out the curve quite satisfactorily and after a few cycles at controlling temperature, you have to start all over again. Recirculation of the hot gases in the furnace has been most helpful. Perhaps this idea has not been carried far enough? Why not re-circulate a portion of

the exhaust gases from the recuperator instead of using excess pure air and reduce oxygen content thereby to just sufficient for combustion and greatly increase rate of heating and efficiency? Control the ratio of fresh air and spent air automatically to produce furnace atmosphere for minimum scaling no matter how temperature controls are operating and how many zones of control exist. Once the proper furnace atmosphere is determined to produce minimum scaling, it is my opinion that practical methods can be devised for controlling such an atmosphere with the fuels that are most economical to burn. It is hoped that future researches will make this possible. The forging industry will be glad, I am sure, to cooperate in this work in any way that they can be helpful, either through the Drop Forging Institute, or through any of the plants direct.

Written Discussion: By L. B. Pfeil, research metallurgist, Mond Nickel Co., Birmingham, England.

I read the paper on "Scaling of Steel at Forging Temperatures" by Messrs. Jominy and Murphy with the greatest interest and regret that the paper did not reach me in time to permit a contribution to the discussion to be read during the Twelfth Annual Convention of the Society.

The authors rightly stress the serious effects resulting from the scaling of steel at forging temperatures and similar remarks might be made with equal justification regarding scaling at the lower temperatures employed in sheet and plate rolling.

The general question of oxidation of steel has not received sufficient attention and I welcome the authors' paper as a valuable contribution to the study of oxidation. The oxidation of steel is a subject of great complexity, for not only is the system iron-oxygen complex, but the oxidizing reactions between iron and oxygen-containing gases were complicated, particularly when a mixture of several gases was involved.

I continued my earlier work on oxidation along the lines of investigation commonly adopted for binary metallic systems, making use of cooling curves, quenching and reheating experiments and the microscope, to determine the various phase fields in the iron-oxygen system over the range Fe_2O_3 to FeO.

Space will not permit more than brief reference to the results obtained. It was found that the melting point of scale increased rapidly and continuously with increasing oxygen content. Oxidation under different conditions as regards temperature, character and rate of supply of oxidizing gas leads to the formation of scale of different average oxygen content. Scale is made up of three phases and a variation of average oxygen content means, in fact, a variation in the relative quantities of these phases present in the scale. Thus, steel exposed to strongly oxidizing gases forms a scale possessing a surface layer of the ferric oxide phase of very high melting point, while steel exposed to slightly oxidizing gases has only the iron-rich scale phase of low melting point. The authors state, on the twenty-sixth page, that scale formed in carbon dioxide has a higher melting point than that formed in steam, and while I consider that statement to be correct under certain experimental conditions, it was not true under all conditions, for in the cases of both gases the oxygen content and the melting point of the scale is affected by the rate of flow of the gas and with the temperature employed. At rates of flow giving in the

case of both gases the maximum rate of oxidation, I have found that at lower temperatures (900 to 1000 degrees Cent.) carbon dioxide gave the scale of lower melting point. The relation between oxidizing conditions and melting point of the scale is important because in the soaking pits and reheating furnaces it is a serious matter to have the protecting scale melt and leave the bare surface of the metal exposed to the oxidizing atmosphere. Indeed, it seemed to me that when the reheating temperature approached 1370 degrees Cent. it would be advisable to make the furnace gases strongly oxidizing so that a scale of high oxygen content and high melting point might be formed. Although a strongly oxidizing atmosphere would cause more rapid oxidation than a weakly oxidizing atmosphere while the protecting scale remained, a weakly oxidizing atmosphere might oxidize the bare metal faster than a strongly oxidizing atmosphere would oxidize through the scale layer.

The authors do not mention the question of blistering, and it may be assumed that they encountered no difficulties in this connection. I have found that blistering of the scale seldom occurred at temperatures over 1000 degrees Cent. especially when fairly strongly oxidizing conditions existed. At lower temperatures, blistering was very troublesome, both from the point of view of obtaining concordant experimental results and from the practical point of view of obtaining a rolled steel product of good surface. Steel beneath an unbroken blister oxidizes much more slowly than beneath an adherent scale layer and as a consequence, an irregular steel surface results, from which it is difficult to produce a good surface finish in the mill.

It is interesting to note that blisters cannot be produced by oxidation in pure oxygen, the reason probably being that no gas is available to support the blisters against the external pressure. The authors' remarks on pages 23 and 30, to the effect that the dilution of carbon dioxide and steam by nitrogen gives the same effect as a reduced pressure of the active gas, are not justified. There is a vast difference between the oxidation of steel in pure oxygen at reduced pressure, and the oxidation in an oxygen-nitrogen mixture with the same oxygen pressure. In the case of both oxygen and carbon dioxide diluted with nitrogen, even with rapid rates of flow, the nitrogen tends to form an inert film round the specimen, which results in a slower rate of oxidation than in pure oxygen or carbon dioxide at the same partial pressure.

The authors' results on the influence of rate of flow on rate of oxidation are of great interest. At slow rates of gas flow the scale consists wholly of the iron-rich phase which is a solid solution with a considerable range of composition. At slow rates of flow, therefore, it would be expected that a gradual increase in the rate of oxidation would result from increasing the rate of flow of the oxidizing gases. With increasing rates of flow, a stage will be reached when the second phase, richer in oxygen, will form at the surface of the scale and the rate of oxidation will, from then onwards, remain constant in spite of further increase in the rate of flow. It might be expected that the oxidation rate for many gases would be the same when the critical gas velocity was exceeded, that is, when the second oxide phase was present. The authors' Fig. 2 shows this to be the case for carbon dioxide and for air. In the case of steam, however, the maximum rate of oxidation at the given temperature (2300 degrees Fahr.) is much greater than in the cases of carbon dioxide and

air. I would explain this result on the grounds that the scale produced in steam is of a much *less* porous character than that produced in air, and that consequently, there is a greater cross sectional path for the atomic diffusion of oxygen inwards and of iron outwards through the scale. The scale found in steam is a very dense deposit of considerable mechanical strength, but from the authors' remarks on the twenty-fifth page it might be inferred that they would anticipate a slow rate of oxidation with a dense and non-porous scale deposit.

I hope that the authors will extend their investigation to a study of scaling at the lower ranges of temperature employed in the rolling of blackplate.

H. H. ASHDOWN:[1] It was not my intention to discuss this paper. However, I would like to offer my congratulations to the authors for this very excellent piece of work. Personally, I have not carried out research work on this, but I have made very careful observations in the works. It might have been noted that when I was discussing the previous paper on forgings I mentioned a temperature of 2200. That temperature was fixed as a result of very careful observation. After exceeding that temperature it was noticed that scaling increased very rapidly. One further point. When one is using solid fuel to attain an increased temperature, often a steam insert is used, and it is noticed by the use of steam that the scaling increases very rapidly indeed, due in all probability to the decomposition of the steam giving an even rich atmosphere of oxygen in the furnace. Not only is the steel much more quickly oxidized, but, as has already been stated, the soft scale produced sticks to the forging and on working becomes embedded in the forging, causing considerable trouble in the subsequent machining.

I think the authors have presented an excellent paper of practical value.

E. J. P. FISHER:[2] Would Mr. Jominy kindly state as to whether or not the authors found in any of their researches on scaling that straight carbon steels, particularly the lower carbons, will scale more rapidly just above the critical range than they do in the critical range or just slightly below the critical range.

W. E. JOMINY: Since no tests were made below 2000 degrees Fahr., I am unable to answer Mr. Fisher's question.

Authors' Closure

The authors are indebted to Messrs. H. H. Ashdown, A. M. Steever, H. F. Wood and L. B. Pfeil for their valuable contributions to the discussion of this paper. They are glad to be able to state that the research is being continued and that tests are already in process to investigate the relative ease of scaling of the more common forging steels, as suggested by Mr. Wood. They also wish to thank Mr. Wood for his generous offer to cooperate and hope that by this means the laboratory results may more quickly be applied to industrial operations.

Both Mr. Steever and Mr. Pfeil bring up the question of the melting point

[1]Consulting metallurgist and engineer, Y. M. C. A., South Chicago.

[2]Metallurgist, Keystone Steel and Wire Co., Peoria, Ill.

of the various forging scales, and the authors hope to make a study of this phase of the investigation at a later date. They will be very much interested in the results and method of attack used by Dr. Pfeil and trust that he will soon publish this information. Some work on the melting points of oxides was published by Oberhoffer and d'Huart (*Stahl u. Eisen*, Vol. 39, p. 165) in 1919. These investigators report the existence of a eutectic between FeO and Fe_3O_4 which contains about 24 per cent oxygen. They also report a lowering of the melting point from that of FeO with increasing oxygen content to that of the eutectic whose melting point is given as 1200 degrees Cent. (2190 degrees Fahr.). From this point there is a general increase in the temperature of the melting point with increase in oxygen content. At forging temperatures the melting point of the oxide will often affect the rate of scaling as indicated by the results given in this paper and also by Dr. Pfeil's discussion. Further, the melting point of the compound formed between the scale and refractory floor of the furnace may have a decided effect on the rate of scaling, and the life of the refractory as well.

In reply to Dr. Pfeil's question about blistering, it may be stated that no blisters were seen in any of the samples produced. It should be remembered, however, that the method of test consisted in quenching directly into distilled water from the scaling temperature, and that this treatment removed much of the scale. Under these conditions it would be easy to fail to observe small blisters. There were instances in which, due to the type of scale formed, none of the scale was removed. No blisters were observed in these cases.

The authors feel that their statements on their twenty-third and thirtieth pages that the dilution of carbon dioxide or steam with nitrogen produces the same effect as a reduced pressure of the active gas is correct under the conditions of the experiment, and that with the rates of flow employed there would be no blanketing effect due to nitrogen. Since this point was questioned by Dr. Pfeil, it was decided to make a few experimental tests in which the pressure of the active gas was reduced without dilution with nitrogen. Tests were made with carbon dioxide at pressures corresponding to those in the tests which were made with 5.1 per cent and 14.6 per cent carbon dioxide and the remainder nitrogen as presented in Table XIII. The rate of flow was kept constant at 60 feet per minute and the temperature at 2300 degrees Fahr. as was also the case in the tests given in Table XIII, the only difference being that the pressure was kept reduced by a vacuum pump rather than by dilution with nitrogen. The following table shows the results obtained by this method and also the results obtained from tests with similar pressures as taken from Table XIII.

As may be seen from this table, Samples 613 and 614 were tested using 100 per cent carbon dioxide at a pressure of 36.9 mm. of mercury. The average loss in weight of these two samples was 0.0392 pounds per 100 square inches. The per cent iron in the scale was 77.6. These results check the average for Samples 321 and 329 as given in Table XIII which were obtained with 5.1 per cent carbon dioxide and 94.9 per cent nitrogen, the pressure of the carbon dioxide being 37.9 mm. of mercury. Likewise, the average loss in weight due to scaling of Samples 615 and 616 obtained with 100 per cent carbon dioxide at 108.3 mm. pressure check the average loss in weight of Samples 322 and 330

Sample Number	Total Time In Furnace. Minutes	Rate of Flow Feet per Minute	Per Cent Nitrogen	Per Cent Carbon Dioxide	Pressure of Carbon Dioxide mm. Mercury	Per Cent Loss in Weight	Average	Loss in lbs. per 100 sq. in.	Average	Weight of Scale in lbs. per 100 sq. in.	Average	Av. Loss in Weight	Av. Wt. of Scale
321	40	60	94.9	5.1	37.9	0.64	0.75	.0305	.0362	.0396	.0468	0.773	
329	40	60	94.9	5.1	38.0	0.87		.0420		.0540			
613	40	60	0	100	36.9	0.80	0.81	.0386	.0392	.0500	.0505	0.776	
614	40	60	0	100	36.9	0.827		.0398		.0510			
322	40	60	85.4	14.6	108.5	1.80	1.78	.0865	.0852	.111	.110	0.777	
330	40	60	85.4	14.6	108.9	1.76		.0840		.108			
615	40	60	0	100	108.3	1.65	1.68	.0790	.0803	.102	.1035	0.775	
616	40	60	0	100	108.3	1.70		.0815		.105			

obtained with 14.6 per cent carbon dioxide and 85.4 per cent nitrogen. The pressure of the carbon dioxide averaged 108.7 mm. The average iron content of the scale from Samples 615 and 616 was found to be 77.5 per cent as compared to 77.7 per cent for Samples 322 and 330 run with nitrogen admixture. These data indicate that with the rates of flow used in making the tests presented in Tables IX and XIII that diluting carbon dioxide or steam with nitrogen produces the same scaling effect as reducing the pressure of these gases.

The possibility that many gases might have the same oxidation rate when the critical gas velocity was exceeded as has been found true of air and carbon dioxide seems, to the authors, rather unlikely. Considering dry air as oxygen diluted with 70 per cent of nitrogen and therefore oxygen with a pressure of 0.21 atmospheres, it happens that oxygen at this reduced pressure and in 40 minutes' time and at 2300 degrees Fahr. produces the same rate of oxidation as does carbon dioxide at full pressure for the same time and temperature. If the conditions are changed by merely changing the time of exposure, all other conditions including the rate of flow remaining constant, then the oxidation rate is different for these two gases as is shown in Fig. 5. Furthermore, the appearance and average composition of the scale formed in air and carbon dioxide were different. Consequently, it appears to the authors that it is merely an accident that under the conditions of the experiment air and carbon dioxide produce the same scaling rate.

PRACTICAL AND THEORETICAL HEAT BALANCE FOR ROTARY FORGING FURNACES

By Theodore F. Schilling

Abstract

The author has made an analysis and comparison of three sizes of rotary forging furnaces using four different fuels. A heat balance is calculated for each of the three sizes considered. The theoretical value determined for the seven-foot furnace is compared with actual data determined on such a furnace. A production rate curve is given, based on the actual versus theoretical fuel consumption. A short discussion is included on the selection and operation of rotary forging furnaces.

ROTARY forging furnaces are now used extensively in all parts of the country. They operate under varying conditions which necessitates their adaptability to the economical heating of a multitude of various types of forgings.

The success of these various installations under such a wide range of uses prompts us to prepare, in detail, a complete analysis of the results that may be expected from any of the standard, well designed rotary forging furnaces.

The following calculations are all based on the assumption that the furnaces are equipped with full automatic temperature control and are operated with a high and low fire in preference to the "on and off" method. This method insures positive control of the furnace atmosphere at all times and results in more economical operation and higher quality finished product.

Heat balances are calculated with four different fuels so that data may be used for the most universal forging conditions. This will also permit a comparison of costs under identical conditions between the four fuels.

Table I shows the four fuels together with their respective heat values and the assumed average cost.

The author, Theodore F. Schilling, is electrical engineer with the Mahr Manufacturing Co., Minneapolis, Minnesota. Manuscript received April 5, 1930.

Table I

Heat Values and Cost of Fuels

Kind	Heat Content	Assumed Cost
Oil	140,000 B.t.u. per gallon	$0.06 per gallon
Artificial Gas	530 B.t.u. per cubic foot	0.70 per M cu. ft.
Natural Gas	1,100 B.t.u. per cubic foot	0.50 per M cu. ft.
Butane Gas	102,400 B.t.u. per gallon	0.08 per gallon
	3,200 B.t.u. per cubic foot	

Fuel Control

When oil is used as the fuel, a full circulating supply system should be provided with sufficient capacity to handle the required demand with a variation in oil pressure not greater than plus or minus one pound pressure per square inch. Recommended oil pressure is twenty pounds per square inch at burners with all valves open. For the last three fuels, namely, artificial gas, natural gas and butane gas, the method of control should always permit any predetermined air-gas ratio to be obtained. The control should also maintain this air-gas ratio regardless of pressure variation in main gas supply line.

Furnace Design

The important features of the furnace design are: method of firing, venting, and general construction of the heating chamber. After these the method of lining, design of structural framework, undercarriage, and drive mechanism should be considered.

The furnace should be over-fired with burners arranged so that part of them fire radially and part fire tangentially into the heating chamber. The vents lead out of the heating chamber at table level and pass up through center of firebrick side walls. This arrangement of burners and vents produces an increased furnace temperature in the direction of the table travel. This insures a thorough heat penetration in the charge when it has reached the point of discharge.

The furnace heating chamber should be designed to burn any of the four fuels without any material changes. The height of the heating chamber should insure complete combustion of gases before products of combustion come in contact with stock. This will prevent flame impingement on stock and produces a constant, uniform gas circulation around the stock. The height of the chamber

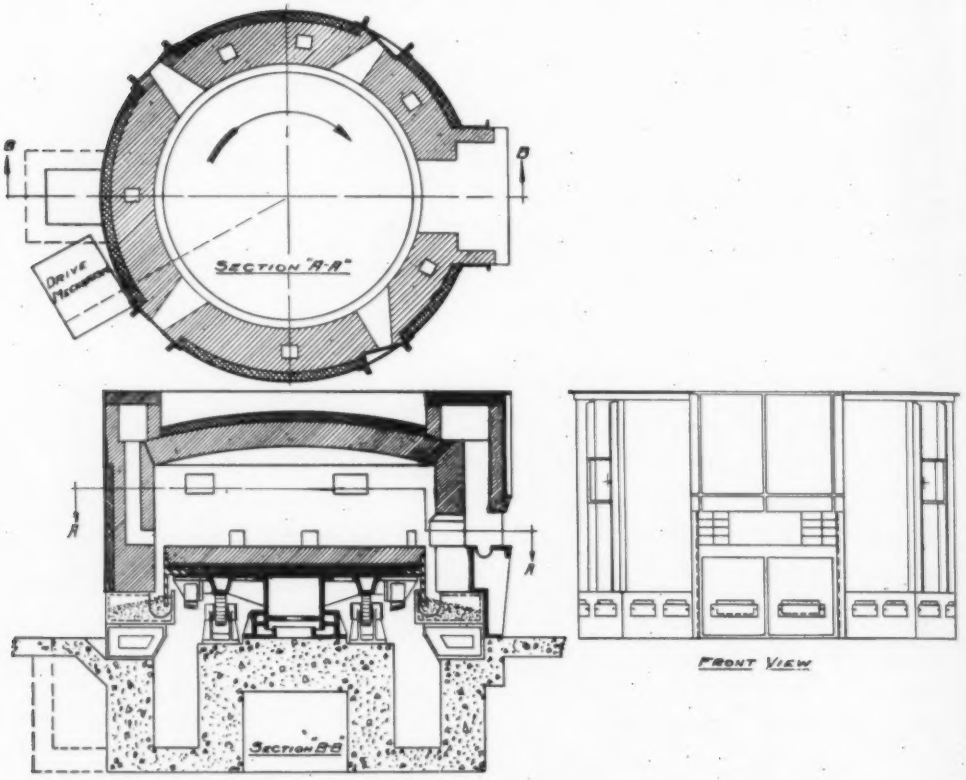


Fig. 1—Diagram of a Typical, Well-Designed Rotary Forging Furnace.

also permits a thorough mixture of gases before venting. This eliminates the dilution of gases (while still burning) with inert flue gases, which is usually experienced in low heating chambers and results in decreased thermal efficiencies.

A practical and economical furnace lining that will result in comparatively low thermal conductivity and low maintenance costs may be obtained with 13½-inch firebrick walls, 9-inch firebrick arch and 7-inch firebrick table. The side walls and arch are insulated with 2½ inches of high grade insulating brick and the table is insulated with 4½ inches of solid insulating material.

The door opening in brickwork should be made large enough to permit a man to enter furnace for repair work. The furnace front shall be provided with a refractory-lined shield forming a flue above door opening through which waste gases from furnace opening may pass. An air curtain pipe should be provided just outside door opening. This arrangement keeps the temperature in front of furnace at a minimum.

The vertical flues in side walls and between refractory shield and furnace proper open into a horizontal, circular, firebrick flue at the top of the furnace, which extends entirely around circumference of side walls. This design will permit all waste gases to be collected in one duct and discharged either through a recuperator or a stack.

The furnace should be provided with steel casing reinforced with structural steel on the outside. Provision must be made to take up thrust of arch on side wall. The rotary table should be of substantial design, supported on roller bearings so that power required to turn table is kept at a minimum. Sand seal castings are attached to the table to close up the opening between table and side walls.

The drive mechanism for revolving the table should be electrically driven through a speed reducer and variable speed transmission. Fig. 1 shows the construction of a typical, well designed rotary forging furnace.

Heat Balance

Rotary forging furnaces are made in innumerable sizes to meet the individual requirements so that an attempt to calculate heat balances for all sizes would require a complete volume. For the sake of brevity, only three sizes will be considered. These have a hearth diameter of 7 feet, 9 feet and 11 feet, respectively, and will handle economically any production between 1500 pounds and 7000 pounds per hour. This range will cover the normal production of the majority of forge shops.

The holding consumption for the three sizes will be theoretically determined after which the heat balance will be calculated. Data obtained from actual tests will be included for comparison. See Table II.

The holding heat of the furnace is that quantity of heat required per unit of time to hold that furnace at the desired temperature under no load conditions. A perfect furnace would be one that would hold the working temperature after once heated indefinitely. This is impossible due to loss of heat through walls, arches, floors and door openings. The holding consumption of these furnaces must, therefore, be established theoretically before a study of the operation of furnaces can be made.

Table II
Furnace Description

Rotary table diameter	7'-0"	9'-0"	11'-0"
Diameter of furnace (inside)	7'-6"	9'-6"	11'-6"
Height of wall (inside)	5'-0"	5'-0"	5'-0"
Radius of spherical Arch	9'-9"	14'-4½"	17'-3⅜"
Thickness of side wall firebrick	13½"	13½"	13½"
Thickness of side wall insulating brick (Nonpareil)	2½"	2½"	2½"
Thickness of arch firebrick	9"	9"	9"
Thickness of arch Insulating Brick (Nonpareil)	2½"	2½"	2½"
Thickness of floor firebrick	7"	7"	7"
Thickness of floor insulation (C-3 Cement)	4½"	4½"	4½"
Door opening	9"x24"	9"x24"	9"x24"

Furnace Areas

Rotary Size	7'-0"	9'-0"	11'-0"
Area—Inside zone of arch:			
h—height of segment	9"	10"	12"
r—radius of sphere	9'-9"	14'-4½"	17'-3⅜"
A—Area = 2 × 3.1416 × r × h	45.9 sq. ft.	75.0 sq. ft.	106.8 sq. ft.
Area of inside vertical wall:			
h—height of wall	5'-0"	5'-0"	5'-0"
d—diameter of furnace (inside)	7'-6"	9'-6"	11'-6"
A—Area = 3.1416 × h × d	117.7 sq. ft.	149.0 sq. ft.	180.7 sq. ft.
Area of furnace floor:			
r—radius of table	3'-6"	4'-6"	5'-6"
A—Area = 3.1416 × r^2	38.4 sq. ft.	63.5 sq. ft.	95.0 sq. ft.
Total inside area of furnace	202.0 sq. ft.	287.5 sq. ft.	382.5 sq. ft.

The working temperature of the furnaces, in all cases, will be maintained at 2300 degrees Fahr. The temperature of the heated charge will be 2200 degrees Fahr. The temperature of the discharged stock in a furnace atmosphere of 2300 degrees Fahr. may be varied above or below 2200 degrees Fahr. by changing the speed at which the table rotates. All furnaces are equipped with variable speed transmissions for adjusting table speed.

Radiation Loss from Furnace

The B.t.u. loss per hour by radiation per square foot of exposed (inside) surface in a furnace with 4½-inch refractory walls and 2½-inch insulation at 2300 degrees Fahr. equalls *6466 B.t.u. per hour.*

Table III gives the values of the constant factor "k" for various thicknesses of refractory and insulation.

Table III
Values for Constant K

Firebrick, Thickness Inches	Insulation, Thickness Inches	Type Insulation	Constant "k"
9	2½	Nonpareil Brick	0.78
13½	2½	Nonpareil Brick	0.63
7	4½	C-3 Cement	1.40

The heat losses through the door openings must also be included and may be considered as black body radiation.

Radiation Through Door Openings

Door openings in furnaces are 24 inches wide with a mean height of 9 inches or a total of 1.5 square feet of opening per furnace. Loss through an opening 1.5 square feet per hour equals 270,000 B.t.u. of equivalent fuel at 2300 degrees Fahr.

Table IV gives the individual and total heat losses for the three furnaces.

Table IV

Individual and Total Heat Losses for Three Furnaces

Loss Through	Constant "k"	7′ Rotary B.t.u Loss Per Hour	9′ Rotary B.t.u. Loss Per Hour	11′ Rotary B.t.u. Loss Per Hour
9-inch Arch with 2½-inch insulation	.78	227,000	378,500	538,000
13½-inch side wall with 2½-inch insulation .	.63	479,000	607,000	735.000
7-inch floor with 4½-inch C-3 Cement	1.4	347,500	575,000	860,000
Black body radiation through doors		270,000	270,000	270,000
Total		1,123,500	1,830,500	2,403,000

Therefore, each furnace must be supplied with an amount of heat equal to that listed under "total" in Table IV in order to maintain the furnace at working temperature of 2300 degrees Fahr.

Table V shows the equivalent fuel required to maintain the furnace working temperature with the four fuels being considered.

Table V

Equivalent Fuel Required to Maintain the Furnace Working Temperature

Rotary Size	7′-0″	9′-0″	11′-0″
Holding consumption in gallons oil per hour	8.02	13.08	17.2
Holding consumption in cubic feet per hour (530 B.t.u.)	2119	3450	4540
Holding consumption in cubic feet per hour (1100 B.t.u. gas)	1020	1662	2185
Holding consumption in cubic feet per hour (3200 B.t.u. gas) ...,	351	572	751

Actual Holding Consumption Data on Standard 7′-0″ Diameter Rotary Forging Furnace

no Load or Holding Test

The furnace was operated with no load under normal operating conditions to determine the actual fuel required to hold furnace at working temperature. Butane gas was used as fuel, and

Table VI

Actual Holding Consumption Data

Time	Gas Meter Reading	Cu. Ft. Gas	Cu. Ft. Gas Corrected	Furnace Temperature Deg. Fahr.	% CO_2	% O_2	% CO
8:30	54015	—	—	2290	13.6	0.1	—
8:45	54105	90	91.2	2320	13.8	—	—
9:00	54200	95	97.0	2310	13.7	—	—
9:15	54295	95	97.5	2300	13.0	—	0.8
9:30	54375	80	82.5	2300	13.1	—	0.7
9:45	54456	81	83.0	2300	12.9	—	0.8
10:00	54539	83	86.0	2300	13.0	—	0.6
10:15	54624	85	87.5	2300	13.1	—	0.6

Average actual fuel consumption to hold furnace ..... 357 Cu. Ft. per Hr.
Theoretical fuel consumption to hold furnace ..... 351 Cu. Ft. per Hr.
% Difference 1.71%

a furnace temperature of 2300 degrees Fahr. was maintained. The furnace temperature was automatically controlled and Orsat readings were taken to insure that furnace atmosphere was identical with normal operating conditions. Data is tabulated in Table VI.

Heat required to raise steel from 60 degrees Fahr. to 2200 degrees Fahr. equals the specific heat of the steel times the rise in temperature in degrees Fahr. and is equal to 346 B.t.u. per pound. The unit quantity of fuel required to raise one pound of steel from 60 degrees Fahr. to 2200 degrees Fahr. equals

$$\frac{\text{346 B.t.u. Required}}{\text{Number B.t.u. Available per unit quantity of fuel}}$$

The available heat per unit quantity of fuel shall be taken as the temperature of the discharged stock or 2200 degrees Fahr. At this temperature the fuels will have the following available heat content.

	B.t.u.
Heat available per gallon of oil at 2200 degrees Fahr.	60,200
Heat available per cu. ft. (530 B.t.u.) gas at 2200 degrees Fahr.	235
Heat available per cu. ft. (1100 B.t.u.) gas at 2200 degrees Fahr.	465
Heat available per cu. ft. (3200 B.t.u.) gas at 2200 degrees Fahr.	1,400

From these values we can obtain the quantity of each of the four fuels consumed in heating up known charges. These values are given in Table VII.

With the data available which shows the holding heat of the furnace and heat required to raise the various charges to 2200 degrees Fahr., we are able to obtain the theoretical fuel consump-

Table VII

Four Fuels Consumed in Heating Known Charges

Charge Pounds	B.t.u. Required	Gallons of Oil	Cu. Ft. 530 B.t.u. Gas	Cu. Ft. 1100 B.t.u. Gas	Cu. Ft. 3200 B.t.u. Gas
1000	346,000	5.75	1471	744	247
1300	449,800	7.46	1910	965	321
1600	553,600	9.20	2350	1190	395
2000	692,000	11.50	2940	1488	495
2300	795,800	13.20	3380	1710	569
2600	899,600	14.95	3825	1930	641
3000	1,038,000	17.20	4410	2230	741
3300	1,141,800	19.00	4850	2460	815
3600	1,245,600	20.70	5300	2678	889
4000	1,384,000	23.00	5890	2980	989
4300	1,487,000	24.7	6325	3190	1061
4600	1,591,000	26.5	6780	3420	1136
5000	1,730,000	28.8	7360	3720	1235
5300	1,833,800	30.5	7800	3945	1310
5600	1,937,600	32.2	8240	4160	1381
6000	2,076,000	34.5	8820	4460	1480

tion for the furnaces when they operate under normal conditions. As in the previous tables the calculations will be carried through with the four different fuels.

The 7 foot rotary shall be considered first, and the charge in pounds per hour shall vary from 1000 to 3000 pounds. This range will cover all operations for which this particular size would be adaptable. See Table VIII.

Actual Operating Data

In order to obtain actual operating data that would be comparable with the theoretical data, it was necessary to take individual readings from several tests so that various charges could be obtained. The same furnace was used for actual holding consumption data and actual operating data. Normal operating conditions were maintained in all cases. Table IX shows data.

In Fig. 2, the theoretical and actual production curves are

Table IX

Operating Data

Gas Metered Cu. ft.	Gas Volume Corrected	Charge per Hr. Lbs.	Cu. ft. Gas per Lb.	% CO_2	% O_2	% CO	Cost per Ton	Theoretical Cost per Ton	Diff.	% Diff.
567	580	908	.639	13.4	0.2	—	3.20	3.16	+.04	1.26
855	872	2112	.412	13.2	—	0.6	2.06	2.07	−.01	0.48
965	984	2500	.393	12.9	—	0.7	1.97	1.93	+.04	2.07
1044	1066	2720	.392	13.4	—	0.4	1.96	1.88	+.08	4.25
1069	1092	2980	.367	13.0	—	0.6	1.84	1.82	+.02	1.10
1117	1140	3090	.368	12.9	—	0.9	1.84	1.81	+.03	1.65

Table VIII

Theoretical Production Data 7'-0" Rotary Furnace—Furnace Temperature 2300 Degrees Fahr.—Stock Temperature 2200 Degrees Fahr.

Oil Used as Fuel

Charge per Hr. Pounds	Gal. Oil per Hr. for Charge	Gal. Oil per Hr. Holding Furnace	Gal. Oil Total per Hr.	Gallons Oil per Pound	Fuel Cost per Ton
1000	5.75	8.02	13.77	.01380	$1.66
1300	7.46	8.02	15.48	.01190	1.43
1600	9.20	8.02	17.22	.01080	1.29
2000	11.50	8.02	19.52	.00975	1.17
2300	13.20	8.02	21.22	.00925	1.11
2600	14.95	8.02	22.97	.00884	1.06
3000	17.20	8.02	25.23	.00843	1.01
3300	19.00	8.02	27.02	.00820	0.99

530 B.t.u. Gas Used as Fuel

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	1471	2119	3590	3.59	$5.02
1300	1910	2119	4029	3.10	4.34
1600	2350	2119	4469	2.79	3.91
2000	2940	2119	5059	2.54	3.55
2300	3380	2119	5499	2.39	3.35
2600	3825	2119	5944	2.28	3.20
3000	4410	2119	6529	2.18	3.04
3300	4850	2119	6969	2.12	2.96

1100 B.t.u. Gas Used as Fuel

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	744	1020	1764	1.760	$1.76
1300	965	1020	1985	1.525	1.53
1600	1190	1020	2210	1.380	1.38
2000	1488	1020	2508	1.255	1.26
2300	1710	1020	2730	1.188	1.19
2600	1930	1020	2950	1.135	1.14
3000	2230	1020	3250	1.082	1.08
3300	2460	1020	3480	1.053	1.05

Butane Gas Used as Fuel—3200 B.t.u. Per Cu. Ft.—102,400 B.t.u. Per Gallon

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	247	351	598	.598	$3.00
1300	321	351	672	.516	2.56
1600	395	351	746	.466	2.33
2000	495	351	846	.423	2.11
2300	569	351	920	.400	2.00
2600	641	351	992	.382	1.93
3000	741	351	1092	.365	1.82
3300	815	351	1166	.353	1.77

plotted for the 7-foot rotary furnace, where butane gas was used as the fuel.

The theoretical and actual fuel consumption and production curves for the 7-foot rotary, when butane gas was used as the fuel, show that the actual fuel consumption is very favorable in comparison to the calculated theoretical fuel consumption. This

actual data were included in order to verify the method employed in making the theoretical calculations. From the results obtained by the comparison of the theoretical and actual fuel consumption for the 7-foot rotary we are safe in the assumption that our method of calculating the theoretical production is correct and proceed, in like manner, to obtain similar data for the 9-foot and 11-foot rotaries. See Tables X and XI.

These furnaces are similar in design and construction and operate under identical conditions. The results, therefore, will be

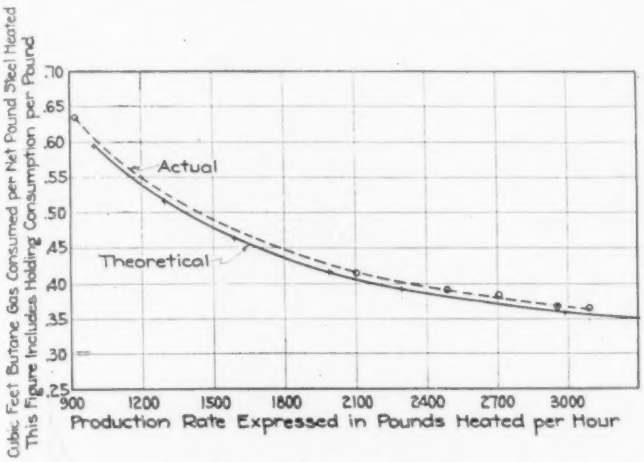


Fig. 2—Theoretical and Actual Production Curves for a 7-Foot Rotary Furnace Using Butane Gas.

proportional, except that holding consumption of the 9-foot and 11-foot rotaries will be changed in accordance with Table V.

Fig. 3 shows the 7-foot rotary installation that was used for making these actual tests.

The theoretical production data for the three furnaces operating with the four fuels gives comparable results and shows that when furnaces are operating within normal range of capacity for which they are designed, a net thermal efficiency between 26 and 30 per cent may be expected. This is without the use of recuperators and is a very high efficiency for forging heats. It is also evident from the data that in order to obtain the maximum net thermal efficiency, the charge of each furnace per hour should be stepped up to the point where the fuel consumption per pound of charge heated begins to rise due to the over capacity of the furnace; or to a point where the increased production necessitates the furnace table to rotate at such a speed that stock is not brought up to desired temperature before being removed.

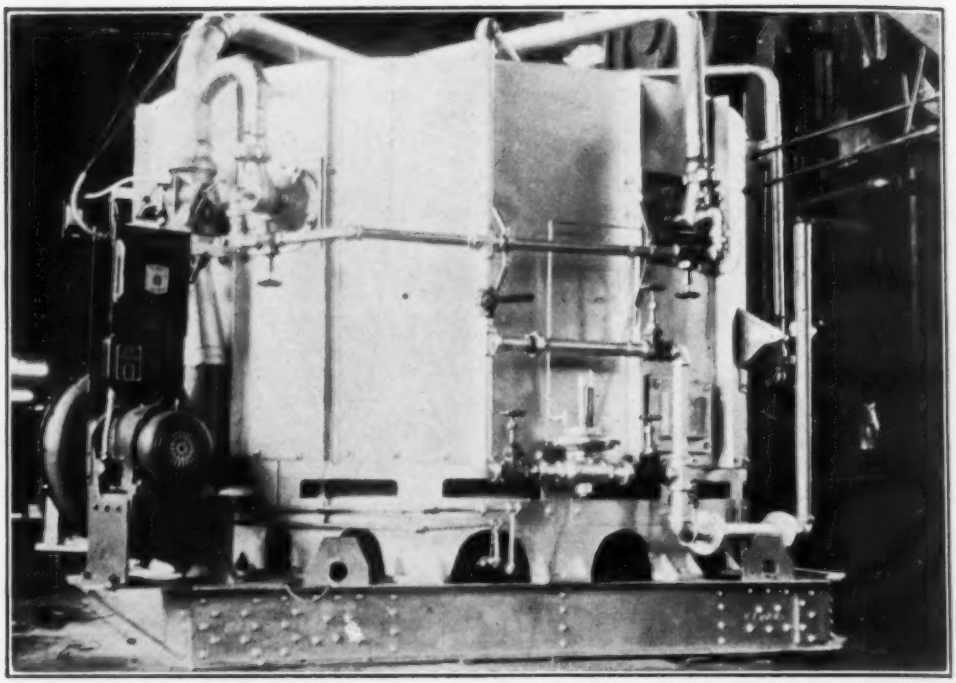


Fig. 3—Photograph of 7-Foot Rotary Installation Used for Making These Tests.

Economics of Operating and Selecting Rotaries

The furnaces should *always* have full automatic control, and this control should be *used* at all times. The furnace atmosphere should be kept reducing to the extent of keeping the scale at a minimum. The ideal method for determining this is by the use of an Orsat for frequent check of the combustion analysis. The importance of this is shown in Table XII, where furnace was operated with a given charge, but under three conditions, namely: excess air, ideal combustion, and excess gas. Butane gas was used as fuel.

These results, while slightly abnormal, are extremely hard to

Table XII

Furnace Atmospheric Condition	Charge per Hr. Pounds	Cu. ft. Gas per Hour Corrected	% CO_2	% O_2	% CO	Gas per Pound Charge	% Increase in Fuel Consumption due to Incorrect Furnace Atmosphere
Ideal	2500	960	13.2	—	0.6	.388	—
Excess Air	2500	1142	12.4	1.3	—	.450	16%
Excess Gas	2500	1385	11.6	—	2.2	.553	35%

Table X

Theoretical Production Data—9′-0″ Rotary Furnace—Furnace Temperature 2300 Degrees Fahr.—Stock Temperature 2200 degrees Fahr.
Oil Used as Fuel

Charge per Hr. Pounds	Gal. Oil per Hr. for Charge	Gal. Oil per Hr. Holding Furnace	Gal. Oil Total per Hr.	Gallons Oil per Pound	Fuel Cost per Ton
1000	5.75	13.08	18.83	.0188	$2.27
1300	7.46	13.08	20.54	.0158	1.89
1600	9.20	13.08	22.28	.0139	1.67
2000	11.50	13.08	24.58	.0123	1.47
2300	13.20	13.08	26.28	.0114	1.37
2600	14.95	13.08	28.03	.0108	1.30
3000	17.20	13.08	30.28	.0101	1.21
3300	19.00	13.08	32.08	.00970	1.17
3600	20.70	13.08	33.78	.00938	1.13
4000	23.00	13.08	36.08	.00903	1.08
4300	24.70	13.08	37.78	.00878	1.05
4600	26.50	13.08	39.58	.0086	1.03

Artificial Gas—530 B.t.u. Used as Fuel

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	1471	3450	4921	4.921	$6.87
1300	1910	3450	5360	4.130	5.78
1600	2350	3450	5800	3.630	5.08
2000	2940	3450	6390	3.190	4.46
2300	3380	3450	6830	2.970	4.15
2600	3825	3450	7275	2.800	3.92
3000	4410	3450	7860	2.620	3.67
3300	4850	3450	8300	2.510	3.52
3600	5300	3450	8750	2.430	3.40
4000	5890	3450	9340	2.330	3.26
4300	6325	3450	9775	2.270	3.18
4600	6780	3450	10230	2.220	3.11

Natural Gas—1100 B.t.u. Used as Fuel

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	744	1662	2406	2.406	$2.40
1300	965	1662	2627	1.940	1.94
1600	1190	1662	2852	1.780	1.78
2000	1488	1662	3150	1.575	1.58
2300	1710	1662	3372	1.465	1.47
2600	1930	1662	3592	1.380	1.38
3000	2230	1662	3892	1.295	1.30
3300	2460	1662	4122	1.250	1.25
3600	2678	1662	4340	1.205	1.21
4000	2960	1662	4622	1.155	1.16
4300	3190	1662	4852	1.130	1.13
4600	3420	1662	5082	1.105	1.11

Butane Gas Used as Fuel—3200 B.t.u. Per Cu. Ft.—102,400 B.t.u. Per Gallon

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	247	572	819	.819	$4.09
1300	321	572	893	.686	3.43
1600	395	572	967	.604	3.02
2000	495	572	1067	.533	2.67
2300	569	572	1141	.495	2.48
2600	641	572	1213	.466	2.33
3000	741	572	1313	.438	2.19
3300	815	572	1387	.420	2.10
3600	889	572	1461	.406	2.03
4000	989	572	1561	.391	1.95
4300	1061	572	1633	.380	1.90
4600	1136	572	1708	.370	1.86

Table XI

Theoretical Production Data—11'-0" Rotary Furnace—Furnace Temperature 2300 Degrees Fahr.—Stock Temperature 2200 degrees Fahr. Oil Used as Fuel

Charge per Hr. Pounds	Gal. Oil per Hr for Charge	Gal. Oil per Hr. Holding Furnace	Gal. Oil Total per Hr.	Gallons Oil per Pound	Fuel Cost per Ton
1000	5.75	17.2	22.97	.0230	$2.76
1300	7.46	17.2	24.66	.0190	2.28
1600	9.20	17.2	26.40	.0165	1.98
2000	11.50	17.2	28.70	.0144	1.72
2300	13.20	17.2	30.40	.0132	1.59
2600	14.95	17.2	32.15	.0124	1.48
3000	17.20	17.2	34.40	.0115	1.38
3300	19.00	17.2	36.20	.0110	1.32
3600	20.70	17.2	37.90	.0105	1.26
4000	23.00	17.2	40.20	.0101	1.21
4300	24.70	17.2	41.90	.00975	1.17
4600	26.50	17.2	43.70	.00950	1.14
5000	28.80	17.2	46.00	.00920	1.10
5300	30.50	17.2	47.70	.00900	1.08
5600	32.20	17.2	49.40	.00880	1.06
6000	34.50	17.2	51.70	.00865	1.04

Artificial Gas—530 B.t.u. Used as Fuel

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	1471	4540	6011	6.011	$8.43
1300	1910	4540	6450	4.96	6.95
1600	2350	4540	6890	4.30	6.03
2000	2940	4540	7480	3.74	5.24
2300	3380	4540	7920	3.44	4.82
2600	3825	4540	8365	3.22	4.50
3000	4410	4540	8950	2.98	4.07
3300	4850	4540	9390	2.84	3.98
3600	5300	4540	9840	2.73	3.82
4000	5890	4540	10430	2.61	3.65
4300	6325	4540	10865	2.53	3.54
4600	6780	4540	11320	2.46	3.44
5000	7360	4540	11900	2.38	3.33
5300	7800	4540	12340	2.33	3.26
5600	8240	4540	12780	2.28	3.19
6000	8820	4540	13360	2.23	3.12

Natural Gas—1100 B.t.u. Used as Fuel

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	744	2185	2929	2.929	$2.93
1300	965	2185	3150	2.420	2.42
1600	1190	2185	3375	2.105	2.11
2000	1488	2185	3673	1.835	1.84
2300	1710	2185	3895	1.690	1.69
2600	1930	2185	4115	1.585	1.59
3000	2230	2185	4415	1.470	1.47
3300	2460	2185	4645	1.405	1.41
3600	2678	2185	4863	1.350	1.35
4000	2960	2185	5145	1.282	1.28
4300	3190	2185	5375	1.250	1.25
4600	3420	2185	5605	1.220	1.22
5000	3720	2185	5905	1.182	1.18
5300	3945	2185	6130	1.156	1.16
5600	4160	2185	6345	1.130	1.13
6000	4460	2185	6645	1.105	1.11

Butane Gas Used as Fuel—3200 B.t.u. Per Cu. Ft.—102,400 B.t.u. Per Gallon

Charge per Hr. Pounds	Cu. ft. Gas per Hour for Charge	Cu. ft. Gas per Hour Holding Furnace	Cu. ft. Gas Total per Hour	Cu. ft. Gas per Pound	Fuel Cost per Ton
1000	247	751	998	.998	$4.98
1300	321	751	1072	.825	4.13
1600	395	751	1146	.715	3.58
2000	495	751	1246	.622	3.11
2300	569	751	1310	.569	2.84
2600	641	751	1392	.535	2.68
3000	741	751	1492	.497	2.49
3300	815	751	1566	.474	2.37
3600	889	751	1640	.455	2.28
4000	989	751	1740	.435	2.18
4300	1061	751	1812	.422	2.11
4600	1136	751	1887	.410	2.05
5000	1235	751	1986	.398	1.99
5300	1310	751	2061	.391	1.96
5600	1381	751	2132	.381	1.90
6000	1480	751	2231	.372	1.86

correct without the use of an Orsat, especially in the case of excess gas. Normal operation requires a certain percentage of excess gas and a small percentage increase over that required will increase the fuel consumption very rapidly, and this same condition is not detectable by even experienced operators.

A common fallacy in forge practice is that the scale is automatically kept at a minimum if a generous supply of excess fuel is maintained in the furnace atmosphere. This is far from true as, due to the dissociation of gases at forging temperatures, the scale is at a minimum through only a comparatively small range. In the case of butane gas, this range is when the furnace atmosphere has a CO content of between 0.5 and 1.0 per cent. When CO content is increased or decreased beyond these limits, an excess scale results. This again shows the importance of checking the furnace atmosphere.

Orsat readings are easily made, and if furnace atmosphere is occasionally checked, a more uniform and generally higher quality of finished forging is obtained with a noticeable saving of fuel.

The selection of a rotary furnace for a definite use is of extreme importance. The universal practice seems to be to select a furnace to handle the peak load that is probably reached only a few days during the month, or to select one of sufficient size to take care of future increase in production.

From the foregoing data, it is self-evident that to obtain real, efficient operation, the furnace should operate at normal capacity the greater share of the time. Peak loads and under-capacity pro-

duction can be handled in the furnace by the flexibility of the operating range.

In cases where production is erratic and fluctuates over a wide range, the installation of two small units will be a much better investment than the installation of a large single unit. A careful analysis of the individual problem will frequently show that the installation of two smaller units will not represent nearly the overhead, even with one unit idle fifty per cent of the time, as that of a large single unit which operates at half capacity fifty per cent of the time.

FORGING PRACTICE IN U. S. NAVAL GUN FACTORY

By W. L. Blankenship

Abstract

In the preparation of this paper the author has endeavored to present a general idea of the forging practice as carried out in the U. S. Naval Gun Factory. In this, he has been handicapped to some degree because of the military character of the material produced, but, has made an earnest effort to present a practical paper, the discussion of which may be of some value to the practical and interest to the technical man. In the treatment of some of the subjects and problems frequently encountered in plants engaged in a similar line of forging work, together with the methods and processes evolved for their solution, it is not desired to convey the idea that these problems could not have been solved by other means, they were however, the best thought of the writer at the time and under the conditions obtaining and are the result of many years experience gained in the manufacture of a product that is subject to a most rigid test, both in inspection and service, namely, Naval Ordnance Forgings.

FABRICATION methods and processes used in connection with the manufacture of forgings for ordinance purposes do not vary materially from those used in the production of material for locomotives or other machinery, insofar as actual fabrication is concerned. However, it probably embraces a wider and more diversified field than any one similar activity, covering as it does all types and kinds of forgings from a 50-ton gun jacket to the smallest die forging weighing but a fractional part of an ounce. It utilizes practically all the standard types of steel and forgeable nonferrous metals, including the various standard bronzes, as well as a number of special compositions of both ferrous and nonferrous light alloys.

In addition to the wide and varied character of the forgings designated for ordnance parts, the very nature of the service and the

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unusual requirements puts their manufacture in a class apart from the commercial field and precludes the acceptance or use of any but the very highest grade material, free from the most minute flaws or defects and embodying the best known workmanship and treatment practice. The necessity for the unusual rigidity of specifications, as well as the very strict elaborate inspection methods applied to all ordnance material, may be better understood if one would consider that a combat ship may be put out of action and the crew seriously endangered because of the failure of some apparently unimportant member of an ordnance assembly, which failure in other types of machines or apparatus might mean nothing more than a temporary shut down for repairs or replacement. This feature is further accentuated because of the limited space available to the ordnance designer and the necessity for reducing weights of ordnance component parts to the lowest practicable minimum.

To the mind of most persons unfamiliar with the construction of guns or other ordnance material, a gun of the cannon type is usually visualized as a large gun barrel with trunnions, by which it is balanced in a stand or carriage and few have more than a very vague conception of the accessory apparatus required for its operation. As a matter of fact the forgings for gun barrels for naval or other large caliber gun units is a very important feature, perhaps the most important single item, but represents only a small part of the material involved in the construction of an ordnance assembly. The numerous and in many cases difficult intricate forgings required for gun bushings, plugs, and breech mechanisms for the gun, in addition to the massive carriage, or mount, with its vast complicated recoil system, elevating and training gear, represent a mass of material many times that of the gun. These together with a number of accessory units, such as torpedoes, torpedo tubes, catapults, etc., go to make up the major portion of ordnance forging work.

A large percentage of the heavier parts for mounts and catapults are made as hollow forgings, some of which are pierced and expanded by mandrelling. Others are made into hollow cylinders which are elongated to several times their diameter by the insertion of arbors and drawing in "V" shaped dies. As it is not practicable to describe in detail the scope, problems and methods of fabrication, heat treatment, etc., of but a small percentage of the forging produced, this paper will be confined to a description of the methods and practice

in the production of a few of the more important items, from a forgeman's view point which have presented difficulties either in fabrication, required physical properties, or both. In this category there might be enumerated minor caliber guns, large training rings, roller paths, breech bushings, expansion chambers, bronze jackets or hoops, trunnion bearing rings and small caliber projectiles.

The material more generally used for gun forgings and a large portion of the heavier parts for mounts, catapults and other ordnace units, is a normal 3 to 3½ per cent nickel acid open-hearth or basic electric steel, for forgings for roller paths, ball races, frictionless bearings, gages, etc., chromium-nickel and chromium-carbon special alloy steel are most frequently used. In the nickel steel field the usual chemical compositions range from 0.15 to 0.38 per cent carbon, 0.60 to 0.80 per cent manganese, 3.00 to 3.50 per cent nickel. In the alloy series nickel-chromium alloys are used most extensively. In this group the carbon varies from 0.25 to 0.90 per cent, manganese 0.60 to 0.80 per cent, nickel 2.00 to 3.50 per cent and chromium 0.80 to 1.60 per cent. These compositions are utilized in the manufacture of heat treated gears, structural and large bearing parts. For frictionless bearings and other parts that are subjected to high compression loads, straight high carbon, manganese and chromium-carbon steels are each used in considerable quantities. The straight carbon steels usually range from 0.70 to 1.10 per cent carbon with a low manganese content. The composition of the carbon-manganese steels is approximately 1 per cent carbon and 1.50 to 2.00 per cent manganese. The chromium-carbon grades more generally used consist of S.A.E. 52,100 type and a special Naval Gun Factory alloy. This alloy is a medium high carbon, high manganese and chromium composition. In the nonferrous group, naval brass (Tobin bronze) and several types of aluminum bronze are specified for parts of assemblies that are especially exposed and subjected to corrosive effects of the elements. The aluminum bronze compositions are varied to meet conditions and desired physical or abrasive properties. This class of material ranges from a normally soft ductile type to a very hard bronze which is used for worm gears and some bearing parts, it is capable of a Brinell hardness of 300 to 400 when heat treated. The obtainable tensile properties of the mild type are from 80,000 to 90,000 pounds per square inch ultimate strength, yield point 40,000 to 45,000 pounds per square inch and an elongation of 35 to 40 per cent.

The methods of manufacture of ingot material and the "prenatal history" of all raw material is obviously of interest to the producer of forgings. Though a discussion of ingot production does not properly fall within the scope of this paper, it is quite evident that if mold design, melting, refining, teeming and cooling practice is not correct, high grade forgings cannot be obtained by the most efficient and careful forging or heat treating methods. It has therefore been our practice to maintain close co-operation between the melting division and forging departments, since it must be recognized that only by the exercise of the greatest possible care in each stage of the long series of processes, from the molten metal to the heat treated and machined forgings can the high character of material required for ordnance work be produced.

Raw Materials

The first and most important essential in the production of high grade forgings is unquestionably, the selection and use of suitable high grade raw material. The next most important factor is believed to be the proper heating for the initial and subsequent forging operations. This feature in the opinion of the writer is one that is seldom given the care and consideration that it deserves. It is further believed that the failure to recognize the importance of proper initial heating is the primary cause of more rejected and damaged forgings than any other single factor in the cycle. Many exhaustive post-mortem investigations conducted for the purpose of determining the probable cause of failures of forgings in test or service, frequently fail in its efforts to discover the real cause, due primarily to the fact, that the investigation is usually started after forgings have passed through the heat treating department where most of the evidence tending to show the effect or possible damage done in the initial heating or forging operations, has been destroyed, or, at least obscured. Therefore the usual conclusion is, that either the material was faulty or the heat treatment had been ineffective, and as a remedial measure, efforts are made to introduce corrective measures in the steel-making and treating department, while the fabricating methods are left out of the equation. The failure to connect defective and inferior quality of forgings with the heating and forging practice are more likely to result in the plants where the responsibility and control of forgings and heat treating operations are divided under

two heads. In this case the heating and forging sections usually feel that their responsibility is limited to shaping and forming the material to the proper shapes and dimensions, and in order to speed up production, frequently proceed to drive up furnace temperatures far above the normal heating temperatures of the material without much thought as to what is happening to the metal. In plants where the sole responsibility for production of forgings, including procurement and selecting material, as well as fabrication and heat treatment is under the immediate direction of one supervisor, alibis are usually given scant consideration and a close check is kept on all operations.

As an instance of the tendency to give the speeding up of heating operations more consideration than proper or thorough heating for forging purposes, is evidenced by the fact, that within the last few months the writer has received several letters containing advertising matter on heating equipment, in which was set forth at some length, the advantages to be obtained by the use of furnaces and burners manufactured or sold by the advertisers. The economies that had been effected in certain industrial establishments that had installed the advertiser's equipment, resulted in some cases in nearly or quite doubling their forging production, by reducing initial heating time.

As a means of keeping a close and accurate check on the various operations and work of the operating crews, and in order to more readily determine the probable cause of success or failure of forgings, a record system has in recent years been inaugurated in the Naval Gun Factory which has been found to be of great assistance in checking results from the different operations, as well as making for a more intelligent selection of the material best adapted for especially difficult forgings. By the use of this system the forgeman is given all possible information covering the salient points in the manufacture of each ingot previous to its selection, by means of a record form. This form is shown in Fig. 1.

For especially important or difficult forgings, the examination and investigation for proving the quality of the raw stock is carried still further, previous to its acceptance for the work in hand. In this case the ingot discard is sawed from the Bl end or bottom end of the ingot and examined microscopically for inclusions, pipes or porosity. Passing this test, the ingot is charged, heated and forged. During the heating and forging process a very carefully detailed record is

INGOT RECORD

Furnace.....Heat No.....Total charge.....Lbs. Ingot No.....

ANALYSIS		
Required	Obtained	Slab
C	.....	.....
Si	.....	.....
S	.....	.....
P	.....	.....
Mn	.....	.....
Ni	.....	.....
Cr	.....	.....
Cu	.....	.....
Fe	.....	.....
SiO_2	.....	.....
CaO	.....	.....
MgO	.....	.....

STANDARD MELT PRACTICE

Instructions	Remarks
A	
B	
C	
D	
E	

Color and consistency of slag at tapping time.....

Est. delivery wt.....

Carbon at melt down.....
Ore added.....Mn. added.....
Last ore to tap .....Hrs.....Min.....
Charging time .....Hrs.....Min.....
Melting time .....Hrs.....Min.....
Time refining to tap ....Hrs.....Min.....
Total time of melt .....Hrs.....Min.....
In furnace after finals...Hrs.....Min.....
Held in ladle .....Hrs.....Min.....
Pouring time .....Min. rate.....Lbs.
Aluminum added...Lbs. Furnace....Ladle....
Condition hot spoon test.....
Setting time.....seconds
Condition metal at tap.....
Lay in molds.....
Condition bottom before charging .....after tap.....
Condition of ladle before pouring....After....
Ingot in mold.....Hrs.....Min.....
Approximate stripping temperature .....
Burying time.....Condition.....
Disposition of ingot .....
Date poured .....
.....
Supervisor

Fig. 1—Ingot Record Used at the Naval Gun Factory.

made covering conditions of ingot when received, heating time, temperatures, etc. This record is made in the form of a forging log. (Fig. 2).

After forging work has been completed and the forging has been given a preliminary or normalizing treatment, the material is subjected to further inspection and test, in order to ascertain the effect of the heating and forging operation. This examination is to some extent similar to that given the ingot before heating, though in this case the test slab may in addition to a micro-structure examination and fracture test, be given a course of heat treatment and one or more tensile specimens taken to determine the probable physical properties obtainable, previous to machining and final treatment of the forging. Having obtained this information the heat treatment section can proceed with treatment of all forgings made from this heat. Should the preliminary examination and tests either before or after forging indicate non-suitability of the material for forgings for which it is intended, the ingot or forging are either diverted and used for less important work, or scrapped.

FORGING LOG

Ingot No.....Heat No.....Type and size.....

TEMPERATURES	SURFACE CONDITIONS OF INGOT
When received	When charged .....
When charged	After breakdown .....
Furnace temperature	When drawn to size .....
Time heating	Chipping necessary? .....
Forging temperature	Red: %—E.....U.....M.....
Finishing temperature	

DISPOSITION

Block No.	Dr. and Pc.	Name of Piece	Test No.
.....	.....	.....	.....
.....	.....	.....	.....
.....	.....	.....	.....
.....	.....	.....	.....

As forged. Preliminary treatment .....

Treated .....

Rough machined. Estimated weight of ingot .....

Passed Estimated weight of discard .....

Rejected ..... Total weight of forgings .....

Diverted

Remarks .....

.....

.....

.....

.....

.....

Date ....

Supervisor

Fig. 2—Forging Log Used at the Naval Gun Factory.

That the process of properly breaking down an ingot in the initial forging operation is one requiring considerable skill, care and experience, is well known to all experienced forgemen and that in order to minimize material losses from surface and in some cases deep cracks and tears, the initial forging work must be performed with light strokes or blows over the entire surface area to toughen the skin, before beginning drastic reduction with heavy full strokes of the forging machine. In the production of forgings that are subjected to rigid exacting specifications and test, the amount, direction and rate of reduction, finishing temperature and cooling are all considered of the utmost importance. Especially is this true in cases where the forgings are subject to heavy alternate stresses or shock in more than one direction. In all cases ingot or heavy billet stock should be broken down with a forging machine or hammer of

sufficient power or weight to make the work effective to the center of the section. Otherwise the core will not have the refining effect of forging or kneading, the outer sections will be elongated and thereby set up severe internal stresses and forging pipes that cannot be rectified by subsequent forging. The forging temperatures of ingot material should invariably be as high as the material will safely stand and should be given very thorough manipulation, in order to assure the necessary grain movement so essential to the process of transposing the coarse crystalline irregular structure of a casting into the fine uniform granular mass necessary

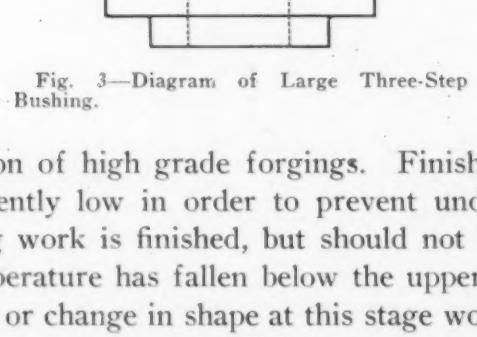


Fig. 3—Diagram of Large Three-Step Bushing.

for the production of high grade forgings. Finishing temperatures should be sufficiently low in order to prevent undue grain growth after the forging work is finished, but should not however, be continued until temperature has fallen below the upper critical range as drastic reduction or change in shape at this stage would tend to break granular cohesion and set up incipient internal cracks. Damage caused in this manner cannot be rectified by heat treatment and will become apparent when the forging is tested transversely to the axis of the major forging operation.

The importance of the amount and direction of the forging operation on material that is subjected to test or service stresses from various angles, was indicated very conclusively in an experience of the writer several years ago, in connection with the manufacture of large 3-step bushings shown in Fig. 3. The specified minimum physical properties of this forging was 65,000 pounds per square inch elastic limit, 95,000 pounds per square inch tensile strength, 18 per cent elongation and 30 per cent reduction of area.

Though the safety factor margin was known to be very narrow, the service stresses had been very carefully calculated, therefore a forging in full compliance with the specified physical properties should have functioned properly. However, several forgings that had passed all of the required tests satisfactorily had failed in service. The first thought was that as they had met all the required physical properties, an error might have been made in the design; nevertheless, it was decided to make an examination and test of the failed forgings before making any changes in design. Two of the forgings were cut into sections and chemical, tensile and Izod specimens were taken. The chemical analysis indicated a normal 0.35 per cent carbon, 3.25 per cent nickel steel; the material was fairly clean and reasonably free from segregation and nonmetallic inclusions. From a microscopic examination it was concluded that the heat treatment had been properly performed and was fairly effective. The results of the tensile test, however, indicated a wide variation in physical properties in the different sections and angles, as well as a considerable falling off in values in the heavier mass sections. In one case the longitudinal test bars indicated physical properties well in excess of minimum specifications; the tangential bars taken near the outer surface barely met the requirements, while those taken from near the center of the section fell off in the elastic limit, elongation and reduction. Radial bars taken normal to the radial axis, were all low in both strength and ductility. The material and structural characteristics of the second forging were very similar to that of the first, though in this case the conditions were reversed, the properties of the tangential bars were in full compliance with specifications, while the longitudinal and radial specimens dropped considerably below properties specified. Retreatment of small sections taken in a longitudinal or radial direction showed but slight improvement and in no case met specified requirements.

After the investigation into the character of the defective forgings a conference was had between the production department, ordnance engineers and the design section; the question was raised as to the practicability of producing forgings of this kind with a homogeneous uniform structure, due to mass effect tending to limit the effectiveness of the heat treatment, one contention being that in order to obtain the required strength and ductility in a forging of this kind, it would be necessary to increase the alloying elements in the steel.

As the design section was not inclined to a change in the class of material and in order to prove or disprove the contention that this forging could not be made to meet the required physical properties with a nickel steel, the writer was given an order for an experimental forging, to be made in such a manner as would permit test specimens to be taken at different angles and at some distance below the surface. From information obtained by the examination and tests made of the defective forgings, the conclusion was reached that as there were no indications that failure was due to faulty material or imperfect heat treatment, it therefore must have been due to some error of commission or omission in heating or in the manner of fabrication, which in one case might be attributed to a lack of forging work in a longitudinal direction and in the other insufficient mechanical work transversely. Acting on this theory a plan of operation was worked out whereby the plastic metal might be subjected to high pressure and a considerable movement in all directions.

A nickel steel ingot with chemical content of 0.32 per cent carbon, 0.71 per cent manganese, 3.09 per cent nickel and 0.28 per cent copper, approximately the same analysis as that of the defective forgings, was selected. The ingot was carefully and thoroughly heated and drawn down to a dimension slightly less than the smallest diameter of the forging. Sufficient stock for the forging blank was cut from the Bl or bottom section, reheated and upset into a counter-bored forming die (Fig. 3) until the two smaller diameters were formed. A ring, comparable with the largest diameter was then placed on top of the die and forging continued until the tool was compactly filled and the forging formed. Without removing from the die, the center was pierced with a small tapered punch and followed by larger punches until the hole was expanded to the desired size. The displacement of material from the hole thus formed, elongating the forging, causing it to project from both ends of the die, when it was again subjected to the full force of the forging press, thereby reversing the flow of metal and shifting it in the direction of the opening in center of the forging. The forging was then put through a very careful heat treatment process, consisting of normalizing at 1650 degrees Fahr., reheated and quenched in a light emulsion bath from a temperature of 1550 degrees Fahr. and tempered by heating to 1210 degrees Fahr. and

furnace cooling. When tested the physical properties obtained from longitudinal specimens were:

Elastic Limit Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Elongation %	Reduction of area %	Fracture
72,300	103,000	20.00	52.68	.5 cup
71,300	103,900	21.30	51.30	.5 cup
77,400	104,900	20.00	49.60	⅝ cup

The values from tangential bars taken from rim of largest diameter and at a depth of 2½ inches from surfaces of the smallest diameters were:

Elastic Limit Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Elongation %	Reduction of area %	Fracture
81,500	104,700	21.50	55.90	.5 cup
78,400	101,800	23.25	57.74	.5 cup
81,500	102,600	23.50	60.81	⅝ cup

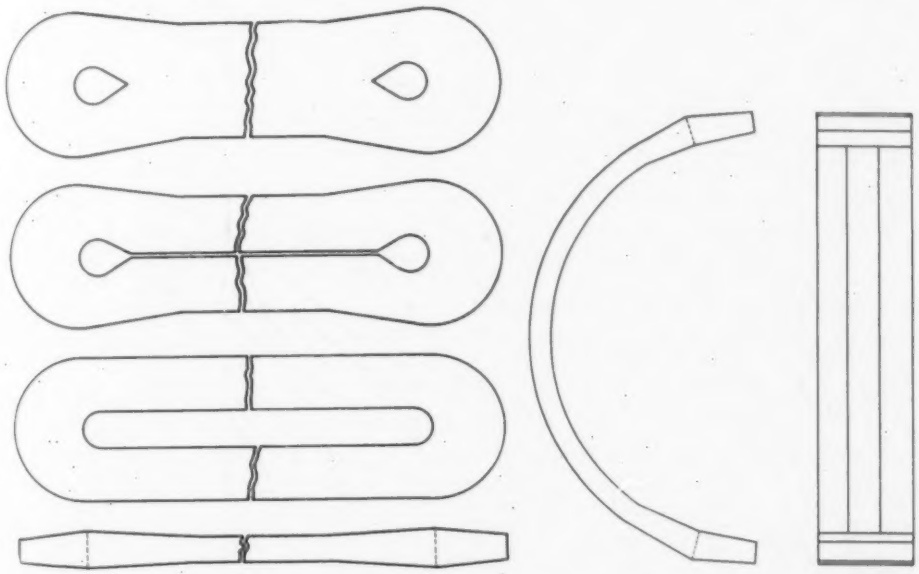


Fig. 4 (Left), 5 (Right)—Sketches Showing Processing of Large Solid Ring Forgings.

All tangential bars were taken more than 2 inches below the outer surfaces and longitudinal specimens 3½ inches from surface. A large number of these forgings have been made in the manner described and where material has been up to the standard, no difficulty has been experienced in obtaining the desired physical properties and none have failed in service. From this and a number of similar experiences, it is concluded that in order to obtain maximum uniform physical properties of heavy heat treated forgings,

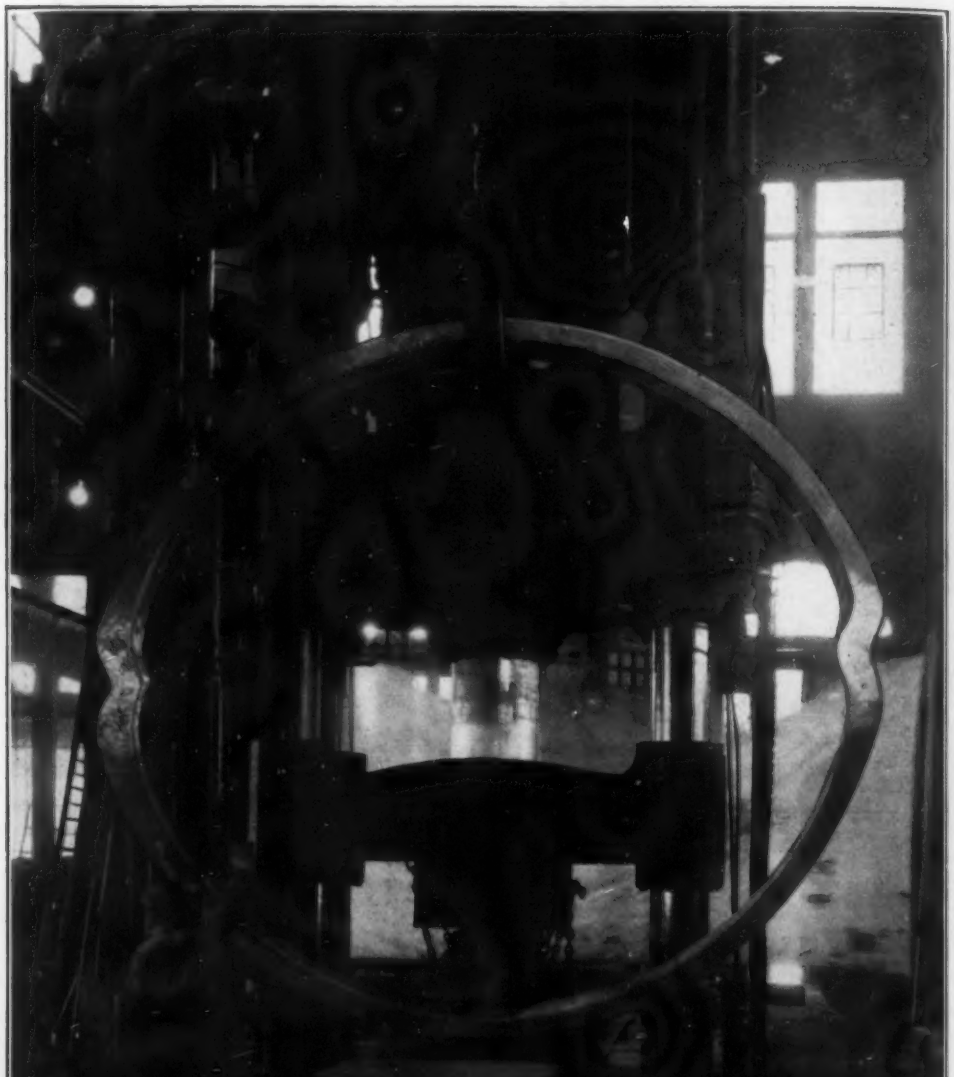


Fig. 6—Photograph of Training Rack Showing Opening Operation.

that the matter of forging, displacements and compression of the metal while in the plastic state is a very essential factor.

The large solid ring forgings for training gears and roller paths, the processing of which is shown by sketches, (Figs. 4 and 5) was made the subject of a paper by the writer and read before the

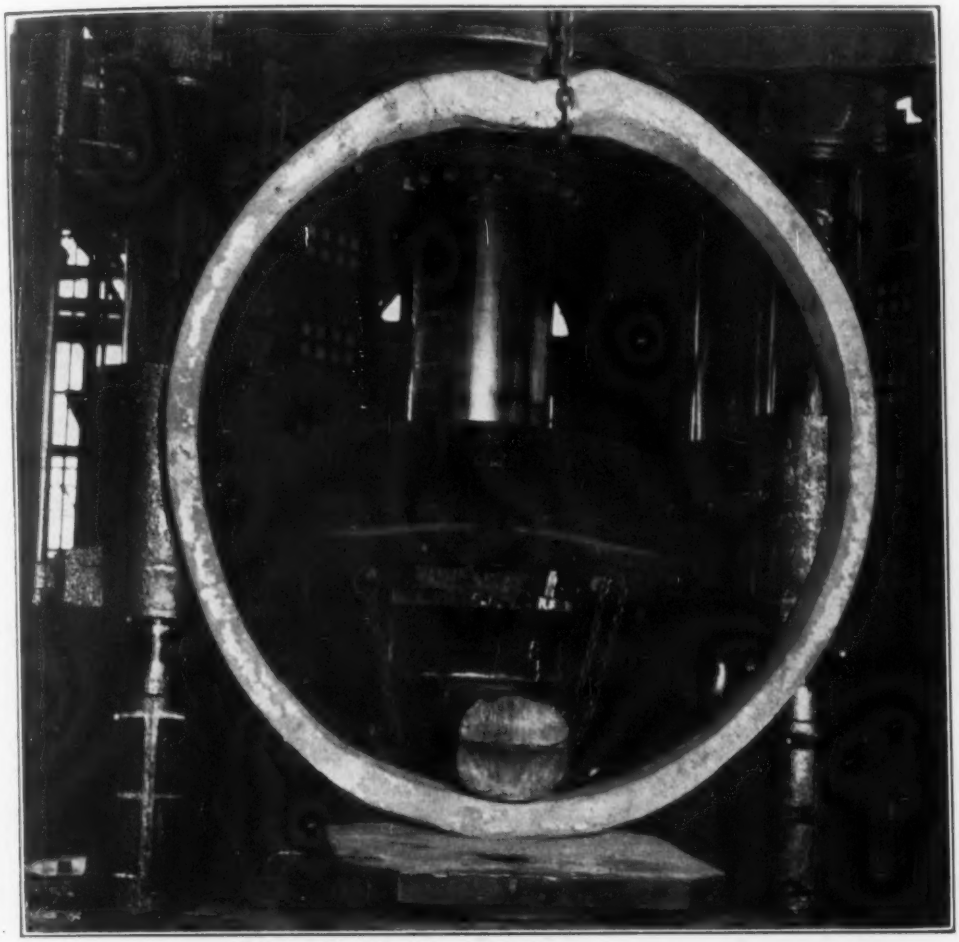


Fig. 7—Forging of the Ring Following Opening Operation.

Washington-Baltimore Chapter of the A. S. S. T. and published in the October 1925 issue of TRANSACTIONS. The problem in this case was more a matter of devising and developing a plan to overcome the difficulty of fabricating solidly forged rings of this size with the limited facilities and equipment of an average forge shop, than the quality of material, heating, or the application of mechanical work. The rather unusual physical properties required for the bearing rings necessitated the development and use of a material of a somewhat unusual composition, in order that the desired degrees of hardness might be obtained by an air cooling and tempering process. To quench a ring of this size and comparative light section would require quenching facilities of an extraordinary character.

The minimum physical properties required in the bearing ring

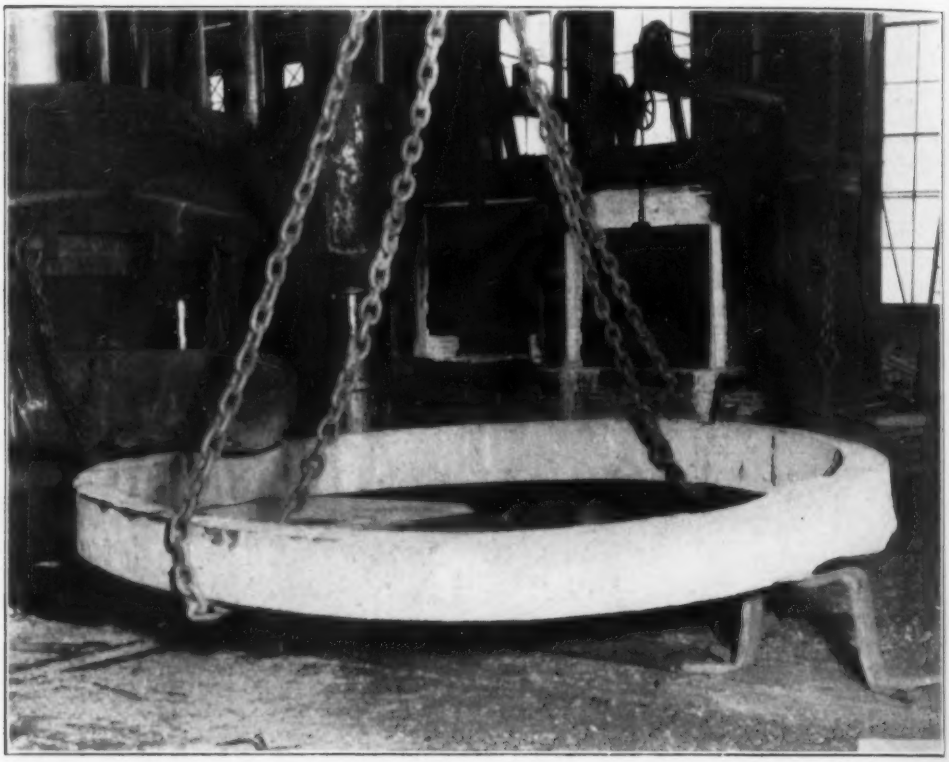


Fig. 8—Forging of the Ring by the Use of a Projecting Beam.

forgings was an elastic limit of 100,000 pounds per square inch, elongation 15 per cent and scleroscope hardness of 45 to 55 or about 320 Brinell. Since writing the paper descriptive of the methods of manufacture of these rings, the sizes and weights, however, have been increased. The composition of the material used for these rings has not been changed materially from that described in 1925, i. e., for the training gear rings a normal 3 to 3.50 per cent nickel steel; for the roller paths, a 3.50 per cent nickel, a 1.40 to 1.60 per cent chromium alloy is used. The forging processes applied in fabrication are approximately the same as described in the original paper; operations consisting briefly of the formation of an eye bar of the proper length and dimensions, splitting between the eyes with an acetylene torch, bending or curving into a semicircle, heating the ends in separate, adjacent furnaces and opening by holding one side under a beam projecting from the forging press and lifting the other with an overhead crane, in a manner similar to that of opening the jaws of a steel trap as shown in the photograph, Fig. 6.

After opening, the heavy irregular sections which constitute the

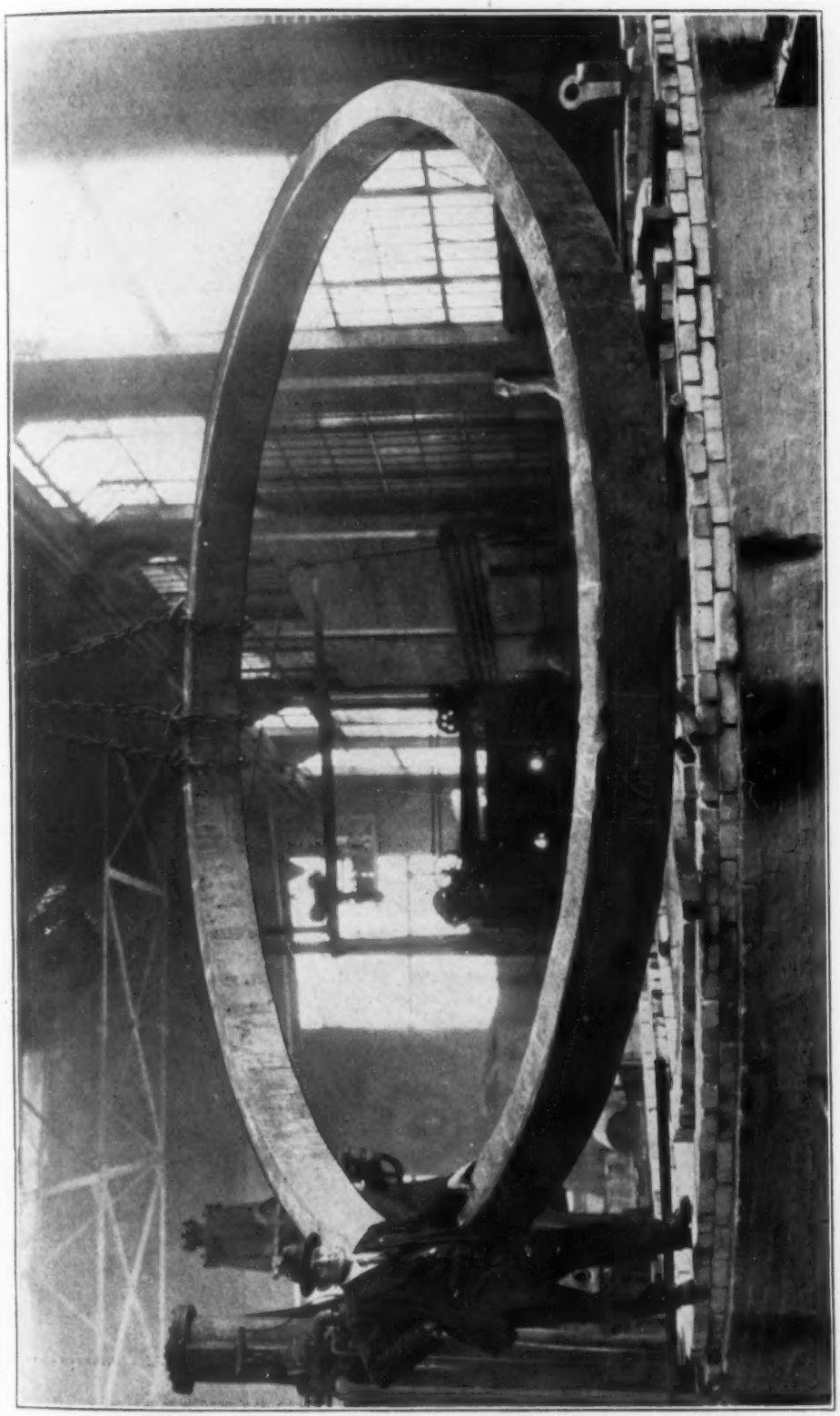


Fig. 9.—Photograph of Forging for Training Gear.

ends of the eye-bar are heated in a small sectional improvised ground furnace and forged by the use of a projecting beam attached to the movable part of the forging press, as shown in Figs. 7, 8 and 9. The manner of heat treating has not changed from that described in the original paper, except that the circular furnace has been somewhat improved and that the air cooling process for hardening the bearing rings is accomplished more efficiently, as the sectional top covering can be removed in a shorter space of time. The degree of hardness obtainable by this process ranges from 60 to 70 scleroscope or about 360 to 380 Brinell, which is modified by a tempering operation.

The specific treatment, with physical test results of a typical roller path ring forging having a composition of 0.41 per cent carbon, 0.51 per cent manganese, 3.51 per cent nickel, 1.57 per cent chromium and 0.26 per cent copper is as follows. After forging was completed it was normalized by heating to 1550 degrees Fahr. and held for three hours and air-cooled by removal of furnace top to 1000 degrees Fahr. Reheated to 1250 degrees Fahr. furnace-cooled. When rough-machined it was hardened by heating from 1500 to 1520 degrees Fahr. and air-cooled, the indicated hardness ranging from 65 to 70 scleroscope. It was then tempered by heating to 1220 degrees Fahr. and furnace-cooled, scleroscope readings taken at 16 points on the periphery ranged from 47 to 52 with an average of 49 for 80 readings. Tensile bars taken from a full size coupon section, treated with ring indicated physical characteristics of elastic limit 100,700 pounds per square inch, tensile strength 38,500 pounds per square inch, elongation 19.30 per cent and reduction of area 55.70 per cent.

The manufacture of expansion chambers which might be described as hollow cylindrical forgings with one end closed in the form of a bottle neck, is not necessarily an unusual or an especially difficult forging to make, though the specifications are very rigid and exacting, and forgings are subjected to a very rigid test and examination. The material specifications for these forgings is a nickel steel and the required physical properties are: elastic limit 65,000 pounds per square inch, tensile strength 95,000 pounds per square inch, elongation 18 per cent and reduction of area 30 per cent, in a transverse or tangential test. The forge dimensions of these parts are approximately 30 inches outside dimensions, 20 inches inside dimensions and 8 feet long, with one end necked down as

shown in Fig. 10. The first series of these cylinders manufactured were made by punching the core from a short heavy cylinder, expanding the inside diameter to the finished inside dimensions and elongating by the insertion of a mandrel in the hole and reducing the wall thickness by means of "V" shaped dies in the forging press. When elongated to the length desired, one end was heated and necked down and contracted to the proper contour by the use of rough formers. As considerable difficulty was encountered in ob-

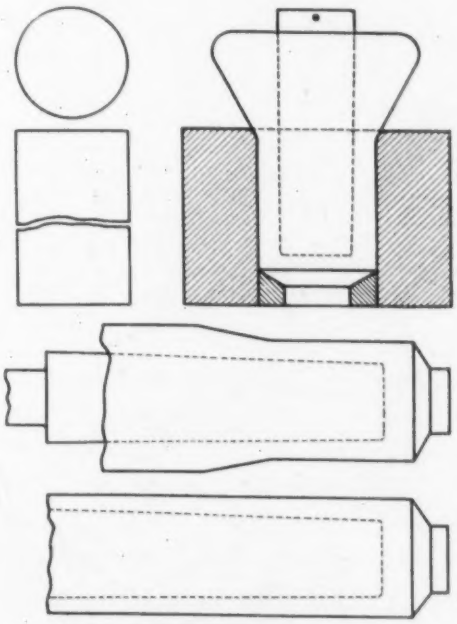


Fig. 10—Sketches Showing Process of Manufacture of Expansion Chambers.

taining the proper contour by forging, as well as a tendency to form crimping seams, due to the drastic reductions in diameter, a plan of operation was evolved by which the constricted section of the finished product is left solid and the small hole bored in the machining operation. This process not only solved the problem but was found to be a much safer and more efficient method of forging, without materially increasing the difficulties of elongation, or cost of machining.

In lieu of forming a straight hollow cylinder and reducing the end, a solid cylinder containing the proper amount of stock is forged to slightly less than the desired outer diameter of the finished forging, dropped into a former as shown in Fig. 10. It is upset to

the approximate outside diameter of the former, and then pierced to the proper depth and expanded to the desired inside diameter, after which a mandrel is inserted in the opening and the piece forged in "V" shaped dies until it is elongated and the wall reduced to the desired dimensions in the same manner as was done with the open end cylinder. In this way the walls are given the necessary mechanical work and the closed end is subject to considerable mass movement and compression while confined in the forming die, thus forming a fine close grained homogeneous structure, capable of producing the desired physical properties when properly heat treated. The usual heat treatment given this forging consists, normalizing by air cooling from 1650 degrees Fahr., annealed at 1450 degrees Fahr. and furnace-cooled previous to rough machining, after which it is quenched in water or light emulsion composed of about 90 parts water, 7 parts mineral oil, 3 parts vegetable oil and sufficient alkali to emulsify the mixture from a temperature of 1560 degrees Fahr. and drawn by heating from 1050 to 1220 degrees Fahr.

Forging Aluminum Bronze

The manufacture of forgings of aluminum bronze for protecting certain types of guns from the corroding influences of salt water, is probably somewhat unusual as regards the manufacture of ingots of required dimensions, forging practice and the required physical properties in forgings of material of the nonferrous group. The largest forgings which are made in the form of hollow cylinders or sleeves, are approximately 6 feet long, 18 inches inside diameter and 22 inches outside diameter. The chemical specification for this material provides for an aluminum content of 7.00 to 9.00 per cent, iron 2.5 to 4.5 per cent, tin 0.5 per cent max., impurities 1 per cent max. The ingots are cast in tapered corrugated cast iron chill molds, large end up and have a mean diameter of about 25 inches and weigh with their hot tops about 16,500 pounds. Previous to forging, a machine cut is taken from the surface of the ingot by means of a special fixture for the purpose of removing scabs and scurf. They are heated in an ordinary heating furnace, but very carefully and slowly to a temperature of about 1680 degrees Fahr. The initial forging operation is carried out in practically the same manner as that used in forging steel ingots, work being continued until the diameter is reduced to approximately 60 per cent of the original

sectional area of the ingot. It is allowed to cool and blanks of the desired lengths for the forgings are cut with a cold saw. These blanks are reheated and upset to about four feet in length and about twice the forged sectional area and are then dropped into a supporting bolster or die and a core about 10 inches in diameter is punched out. This operation usually results in tears which have to be machined out before continuing forging. After the laps or tears have been machined out the inside diameter is expanded to the desired size by rotating on an arbor and finally elongated by means of the insertion of an arbor and reducing the wall thickness in "V" dies. Because of the narrow margin between the proper forging temperatures and fusing point of the material and the narrow forging temperature range, the mechanical work must be performed rapidly and necessitates frequent heating. This heating must be carefully performed as any over-heating or drastic mechanical manipulation at too low a temperature materially affects the structure and the attainable physical properties. The physical properties of these forgings are: yield point 37,500 pounds per square inch, tensile strength 75,000 pounds per square inch, and elongation 30 per cent. The desired properties in forgings of light sections are sometimes obtained without heat treatment, though the heavier sections must usually be given a course of treatment, consisting of a quench and a low tempering heat.

This material responds quite readily to heat treatment operations and the treatment found most effective and effecting the best results for forgings of the sections described in the above is, quenching in water from a temperature of 1680 to 1720 degrees Fahr. and tempering from 700 to 750 degrees Fahr. The average properties obtained from a number of these forgings, yield point 42,500 pounds per square inch, ultimate tensile strength 85,600 pounds per square inch, and elongation of 40.6 per cent.

Bearing rings used in the construction of frictionless rolled bearing units are made up of an inner and outer ring with a double set of rollers between. Such bearings are subjected in service to a very high compression work load and must withstand very severe shocks. The problem in their manufacture is more a matter of obtaining and selecting a material sufficiently strong and hard to carry the applied loads without indenting or breaking. Several types and compositions of high carbon and alloy steels were tried and discarded before a material with sufficient compressive strength and

deflectibility was found. The material found to more nearly possess all of the desired characteristics and capable of meeting the exacting drastic requirements might be termed a carbon-manganese-chromium alloy. This is a product and a Naval Gun Factory development. It is fairly malleable and when carefully heated and manipulated can be forged without difficulty. Medium and light sections harden readily in oil and at a much greater depth than carbon steels or most other alloys. Uniform heavier sections can safely be quenched in tepid water or light emulsions and it can be tempered at a temperature of 450 to 800 degrees Fahr. without materially lowering its resistance in compression loads and strength. When tempered to the high temperatures it possesses an unusual high degree of resiliency and is very effective in certain types of springs.

In the preparation of billet stock of this composition, the exercise of great care in the heating and initial forging of the ingot is absolutely necessary. It has been found that the best results are obtained by charging the ingot into the forging furnaces, heated and forged as soon as possible after stripping from the mold, rather than burying or cooling in a soaking pit, thereby obviating the cooling hazard until the metal has been reduced to billet or bar form and given a preliminary anneal. Other precautionary measures found to be highly desirable, if not absolutely essential, in subsequent heating and forging are carried along throughout the various fabricating processes, i. e., after heating for forging the material is not permitted to cool until all operations have been completed and the parts have been at least partially annealed. The optimum initial forging temperatures for this material is a maximum of 2075 degrees Fahr. and a minimum of 1550 degrees Fahr., the latter temperature being used for light planishing and straightening only. Immediately after the operation of reducing ingots to billets or bars, to be used as forging stock, the product is charged into an annealing furnace, raised to a temperature of about 1550 to 1600 degrees Fahr. and slowly cooled. During the fabricating processes, temperatures are not allowed to fall below 1100 to 1200 degrees Fahr. outside of the furnace. After mechanical work has been completed, the product is immediately subjected to a normalizing and spheroidizing treatment, and is then ready for machining, final hardening and tempering.

In cases where any great amount of material is removed by machining, or parts are especially intricate in shape, a second anneal-

METALLURGICAL AND TESTING DIVISION
U. S. NAVAL GUN FACTORY
REPORT OF SPECIAL MECHANICAL TESTS

Test Number BB 387. For Plant OH Date 11-17-28
Material Test pieces, of Special Carbon-Chromium Steel, Naval Gun Factory
Specifications Tensile, Compression and Hardness tests.
Testing Machine ..... Operator .....

	Heat Treatment		Tensile Specimen		Compression Disks		
	Quenched	Drawn	T. S.	Elongation	Ultimate Compressive Stress	Elastic Limit	Permanent Set After
No.	°F.	°F.	P. S. I.	% In 2"	P. S. I.*	P. S..I.	Test
3	1480	350	223500	0.00	300.000	240.000	0.00085
5	1480	450	287000	0.00	300.000	240.000	0.00068
9	1480	500	233000	0.00	300.000	250.000	0.00045
11	1480	550	217000	0.00	300.000	230.000	0.00060
15	1480	600	160500	0.00	300.000	220.000	0.00070

*without fracture

	Rockwell Hardness Diamond Cone, 150 Kg. Load "C" Scale			Vickers Hardness Pyramidal Cone 30 Kilos Load 2/3 Obj.		
No.	Min.	Max.	Avg.	Min.	Max.	Avg.
3	60.0	60.7	60.5	722	742	728
5	58.0	59.0	58.5	682	682	682
9	56.4	56.9	56.7	601	653	637
11	55.2	56.7	56.1	626	662	649
15	54.0	54.5	54.3	593	635	616

Fig. 11—Special Mechanical Tests Report Used by the Naval Gun Factory.

ing or drawing operation is considered desirable in order to remove stresses set up by rough machining, also that the piece may be less likely to distort in the hardening process. The treatment for removing strain consists of a slow careful reheating to a temperature slightly below that used in spheroidizing, holding for a time sufficient for a full saturation and furnace cooled. Proper hardening temperature of this material ranges from 1480 to 1540 degrees Fahr. depending on sizes and sections to be treated. For work requiring the maximum abrasive hardness and compression strength, the tempering temperature range is from 450 to 550 degrees Fahr., for removing quenching strains and possible brittleness. Obtainable properties for this material by tensile and compression test is shown in Fig. 11.

Manufacture of Shells

The manufacture of shell or projectile forgings is not unusual nor is the process considered especially difficult in a plant equipped with suitable machinery and facilities. It will be remembered by a great many, that during the period of the World War, the demands

of allies and our own government for all kinds of munitions, including projectiles of all calibers was so great that a number of the largest metal working plants of the country went into the manufacture of this class of material on a gigantic scale. They utilized all of their standard facilities and concentrated in some cases their entire engineering staffs on design and development of special tools, presses and other special apparatus, which enabled them to turn out forgings

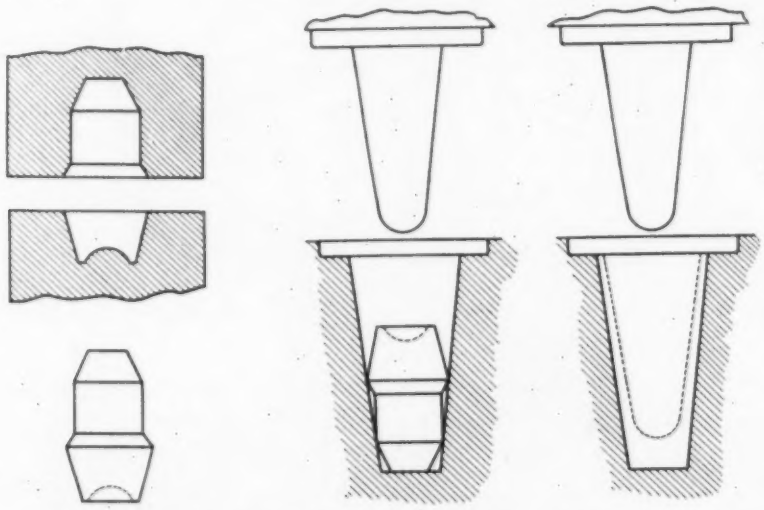


Fig. 12 (Left), 13 (Right)—Sketches Showing Sequence of Operations in Forming Shell Bodies.

of this kind in enormous quantities, efficiently and economically. After the ending of hostilities most of the establishments that had been engaged in the production of munitions, dismantled or scrapped all of their special equipment in order to re-adjust their plants to peace time activities.

The required physical properties for the bodies and plugs was an elastic limit of 75,000 pounds per square inch, tensile strength 105,000 pounds per square inch and an elongation of 20 per cent. As these properties could not normally be obtained with a straight carbon steel and the cost of standard alloy material was in this case prohibitive, the procurement of suitable material at a low cost was the first problem to be solved. After some study and investigation it was found that a medium carbon high manganese steel having a carbon content of 0.45 to 0.55 per cent and 1.00 to 1.30 per cent manganese was obtainable at a cost slightly in excess of straight carbon steel and that it could be readily heat treated to comply with prop-

erties specified. With the material question settled, the solving of the problem of fabricating at a rate and price that had been established when this class of material was being manufactured with special equipment was next in hand. The Naval Gun Factory was not equipped with piercing or drawing presses or other special devices for the production of shells and had never made forgings of this kind other than a few small lots for experimental purposes. Due to the comparatively small quantity required (in this case the

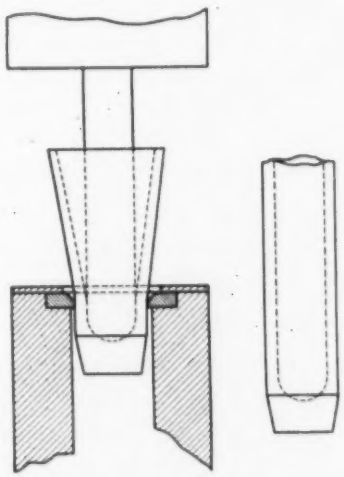


Fig. 14—Diagram Showing Operation of Producing Shells.

installation of special equipment could not be considered) it was therefore necessary to devise methods whereby the work could be consummated by the use of regular forge shop equipment. As in many lines of engineering effort the limitation of available equipment and the absolute necessity for the performance of a task that at first sight appears impossible is ultimately responsible for the development of peculiar, yet effective production methods.

In this case the task resolved itself into a problem of the design of forming tools with which small hollow cylinders with closed end and thin concentric walls could be rapidly turned out by the use of steam drop hammers, the only machines available for the purpose. It was manifestly impracticable to produce these deep pierced blanks as a straight drop forging, as the ratio between the depth and diameter of the cavity was too great to be successfully formed with straight piercing punches. The eventual solving of the problem, development of tools, effect and sequence of operations in the form-

ing of the shell bodies, is graphically shown by sketches, Figs. 12 and 13.

In order to prevent the possible use of unsound material the inspection requires whole examination of the fracture of each projectile blank, therefore the stock must be broken up rather than cut or sawed; in this case bars were nicked in a turret lathe by means of diamond pointed tools and broken under a small steam hammer. The forging operations were performed by the use of three drop hammers of a rated capacity of 3500-pounds, 5000-pounds and 8500-pounds. As may be noted the first forging operation consisted of shaping up the billet in dies for the initial piercing process as shown in Fig. 13.

The next step was to pass the blank through a heating furnace to the second die where it is pierced and flared to a shape resembling a flower pot, after which it was passed to the heavy hammer and forced through the drawing die, the cavity closed, sides straightened and the piece elongated to the desired length of the finished forging, the powerful heavy hammer being used in most cases as a drawing press. The weight of the ram and die together with the force exerted by steam pressure on the piston was sufficient to push the smaller forgings through reducing die; for the heavier shells a few short stroke blows served to complete the operation. Shown in Fig. 14.

The matter of identification marks and record of manufacture, treatment, and test of forgings is probably carried to greater length in this plant than is considered necessary by most other manufacturing establishments. In addition to the name, drawing, and piece numbers of each forging or lot of small forgings is given a heat or serial number which is carried as a permanent record in the forge, treatment, and testing divisions. These numbers are stamped on the forging at the time of manufacture and are carried along through the various finishing and machining processes and into the service. This record is maintained during the life of the assembly of which the forging is a component part and consists of the initial heat number, analysis, pouring, and forging records together with heat treatment and physical properties. It is also cross-filed in such a manner as will readily permit identification of all parts manufactured from any heat or ingot.

The object and desirability for the maintenance of this record may be readily seen when it is considered that the material is used on ships, on stations, and various parts of the world and questions and

inquiries regarding the functioning of parts could not be understood or intelligently handled without some record of this kind.

In the foregoing chapters the author has endeavored to bring out a few of the many problems and difficulties encountered in the manufacture of forgings of a wide and varied character, together with the methods adopted for their solution; this paper is, therefore, intended to cover the practical side of the manufacture of forgings rather than an attempt to disseminate results of research work in connection with metal manufacture or heat treatment technique and is dedicated to practical forge and heat treaters and others interested in this particular line of work.

DISCUSSION

Written Discussion: By H. H. Ashdown, consulting metallurgist and engineer, Y. M. C. A., South Chicago.

To supervise the manufacture of the multivarious products, their broad range and character calls for a man of wide experience to deal successfully with the present day demands for Naval Ordnance and the author has well filled an unenviable position.

For successful production, not only in Naval Ordnance but in many other fields, co-operation of the three essential departments,—steel-making, forging and heat-treatment is imperative.

The first and most important essential in the production of high grade forgings, as outlined on the fourth page, is the making of high quality ingots. The suggestion in the paper of the selection of high grade raw materials, although of advantage is no guarantee of this, The method of manufacture and subsequent handling of the liquid steel are the greater controlling factors. One sees little advantage of a full melting log being supplied to the forgeman unless he be an experienced steel-maker; even then it may be of little value. The complete record is best kept by the laboratory and the responsible metallurgist should be more competent to decide on the fitness of ingots for any prescribed purpose. The most useful information for the forge is that subsequent to tapping,—casting temperature, rate of teeming, conditions in the mold, time of stripping, general appearance of ingot surface and, most essential, pit sample analysis.

Although the care necessary in the initial heating of large ingots or billets preliminary to forging is of vital importance even to the extent of preliminary steam heating, it is at the other end of the operation,—the question of "cold forging" that requires equally much, if not more attention as it is from that end that perhaps more failures arise. Disastrous results to many valuable forgings have been definitely traced to this cause. The temperature range in which forging operations should be effective, as referred to on page 8, requires much clarifying.

The maximum temperature to which ingots and billets should be heated

for forging depends on their composition and general characteristics. For instance a soft maximum forging temperature around 2200 degrees Fahr. suitable for nickel-chromium-molybdenum and high carbon steels as employed for Naval Ordnance would be disastrous if employed for high percentage nickel steels but these conditions may safely be followed for Ordnance nickel and low carbon steels.

The time to stop forging work is dependent on the class of material and cross-section of the forging, as the internal defects referred to on the eighth page are mainly the result of tension stresses which in small sections are less pronounced. This point in alloy steels is strongly indicated by the marked resistance to deformation at a red heat, at a temperature around 1600 degrees Fahr. by the solidity of the blow of the hammer and at the press, by considerably increased indicated pressure and for hollow work, growl on the mandril.

Little attention need be paid to this. Any grain growth that might result from cooling from a high finishing temperature would be corrected by subsequently subjecting the forging to a normal heat treatment.

The great risk is small internal contractional ruptures due to low temperature working and one is surprised to note the suggestion that forging should not be continued below the recalescence point. Forging work should cease, well removed from the critical range. There is little advantage in obtaining a superfine structure if the forging is subsequently scrapped.

It is of comparatively recent years that forgemen have been discouraged in their pride of planish finished forgings, the colder the finish the finer the planish. Little wonder under those conditions that highly expensive forgings, both solid and hollow, for Naval Ordnance were rejected as a result of "slip" and "woody" fractures in physical tests, which today happily are more rare than common.

The method of manufacture of the large ring forgings for training gears and roller paths is unique in overcoming what in the past might have been considered the dimensional limitations of the forging press. The only objection one might raise to this method is, in opening out the semicircular forging, considerable stresses must be induced into that half which is bent from the concave to the convex form in the cold condition. It would be of considerable interest to learn if these rings, all costs considered, could not be produced more cheaply in the tyre mill.

Passing on to shell manufacture it is appreciated that during the war any method of satisfactory production was acceptable and for the plant available the methods shown are commendable. The flower pot shape resulting from the first operation recalls the form produced under the hydraulic press some 25 years ago. The press work of today has no comparison. At a rapid rate they are produced from the block almost a perfect cylinder in one operation, the smaller shell being also chambered and finished in one subsequent draw and the large heavy types in two draws.

From a practical point of view and covering so wide a field of operations the paper is of considerable interest and reflects much credit on the author.

Author's Closure

The stenographic report of Mr. Ashdown's oral discussion of my paper, "Forging Practice in U. S. Naval Gun Factory," would indicate that his conception of the method employed in opening these rings is erroneous, doubtless due to the rather meager description of the operation in this paper and without reference to the original paper which was published in TRANSACTIONS of October, 1925.

It would appear that Mr. Ashdown is under the impression that these rings are formed by a method sometimes referred to as the "buttonhole process" consisting of splitting or stamping a slot in a rectangular bar or slab, which is opened by means of tapering drifts or shear dies and finished on arbor or by drop forging. This method is used extensively in forming small rings such as ring gears for automobiles, etc., but could not be used for forming rings 20-feet in diameter and weighing 20,000 pounds.

Following the forging operations in sequence and by referring to sketches, Fig. 4 of this paper, it may be noted that the first operation is to form an eyebar of proper length, slightly less than half the circumference of the ring; the second operation consists of splitting between eyes with an acetylene torch; third, heat ends, open and straighten sides forming a link; fourth, heat and curve to desired radius of finished ring, thus forming a semi-circle. For the sixth, or opening operation, we heat the ends of the semi-circle to a normal forging temperature, placing the lower section equidistant from the ends of the arc, under the end of a heavy beam held by the forging press, the heated ends acting as a hinge, the two halves are pulled open by means of an overhead crane in a manner similar to the opening of a steel trap.

After opening in the manner described the two short, hinge or end sections which have been intentionally made considerably heavier than the rest of the circle are reheated, reduced, and shaped up by means of the beam projected from the front of the forging press, as shown in Figs. 7 and 8 of the paper. None of these operations involve any cold forging or bending.

PROCESS RELIABILITY IN STEEL MAKING

By George A. Dornin

Abstract

In this paper the importance of reliability of steel-making processes is brought out. The unreliability of our present day methods is shown. From basic metallurgy the author arrives at, what he believes must be, the foundations of what steel-making will become if it is to be a process under control and thereby produce reliable metal.

ABOUT ten years ago a group of very earnest metallurgists got in the habit of meeting together to discuss their common problem which was the heat treatment of steel parts to be used in industry. The lives of all of us in one way or another are jeopardized when these parts fail and the importance of achieving reliability is apparent to everyone. These men ultimately formed the American Society for Steel Treating and as they continued their work found that many of their heat treatment problems could be directly traced to bad steel making. The melting session of the American Society for Steel Treating was then inaugurated and has continued from that day a most useful feature of the Society's activities.

It seems eminently fitting therefore that a paper such as I shall attempt to make this one should be read before this body; a paper on process reliability.

It is not claimed that what is said will be the last word on this subject for I know that I have not enough knowledge to make it so, but it is hoped that what is started in this paper will be taken up by others and ultimately bear fruit in changing many of our present unreliable steel-making practices into processes that follow sound metallurgy and that thereby have at least a chance to produce reliability. Direct attention will be called to many of these practices that I believe to be inherently unreliable and an attempt made to point the way to changes that must be brought about to make

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, who is a member of the society, is metallurgical and consulting engineer with the Gathmann Engineering Co., Baltimore. Manuscript received June 24, 1930.

steel-making not an art as it is today but a science as it must be if it is to reliably serve our purposes.

In a booklet written many years ago by Emil Gathmann and the writer we advanced certain fundamental essentials for sound steel. These were:

First—That the bath of metal be thoroughly deoxidized before teeming, so that no chemical reaction can take place during solidification.

Second—That the metal be cast in an ingot mold whose inside dimensions are enough greater at the top than at the bottom to prevent internal bridging.

Third—That the ingot be kept in substantially a vertical position until solidification is complete.

Fourth—That a refractory sink head be used in all ingot moldings.

After many years of practice the writer sees no reason for withdrawing or modifying any of these statements. He believes, however, that the first statement requires much amplification and he will attempt hereafter to add to it. The second statement can stand as originally written. The third statement will possibly have to be modified and completed so that it will take care of flaws brought about by crystal cleavages. The fourth statement is not truly fundamental to sound steel but is only commercially necessary to get steel of this character at not too great a cost.

In a paper read before this Society by the present writer in September 1927 an attempt was made to classify our various steel-making processes according to their ability to make sound clean steel.

We must realize that there are three distinct phases to the melting side of steel-making. These are: First—Getting the charge molten. Second—Oxidizing out of this molten bath detrimental elements and those not wanted in the final product. Third—Deoxidation or the reduction of iron oxides and gaseous oxides present in the bath and the elimination from the bath of these reaction products. Unless iron oxide is reduced from the molten bath reaction must continue during solidification and the process therefore does not meet the first requirement of sound steel-making.

In a paper read before this Society in September, 1929, I called attention to the fact that deoxidation cannot completely take place as

long as the slag covering the bath has any oxidizing effect. I proposed at that time that our open-hearth process be changed so that the finishing of heats could be done under a non-oxidizing slag.

Our present day steel-making processes are composed largely of an accumulation of make shifts. These have come into the industry from two causes. First—Lack of knowledge of the metallurgy or physical chemistry of steel making, and Second—Because steel makers when knowledge due to research has been accumulated often feel that its application is too costly to embody into the process. In the past ten years a vast amount of accurate metallurgical knowledge has accumulated. This is due to the research work of many men among whom I may name C. H. Herty, Jr. and his associates and I think that the time has come to analyze our steel making and apply it along lines that follow the teachings of research. To date little progress has been made in this direction. The great bulk of our steel is still made under processes that give no chance whatever for reliability and I believe that the extra costs such as rejections, chipping, etc., make up a loss total far greater than would be the case were processes soundly carried out on the basis of our present day knowledge, and much too great a loss to have a basic industry such as steel-making handicapped by it. This does not mean that our present day plants must be discarded but only that they must be modified where necessary to enable them to carry out the processes according to sound metallurgy.

As an example of how unsoundly the bulk of our steel is made, I will cite two practices under which more than half of our total steel supply is made today. The two practices to which I refer are the rimming of steel and the killing of steel in the ingot mold, using aluminum or silicon. Neither of these practices completes final reaction before the beginning of solidification and therefore neither practice can produce anything but unreliable steel nor can either practice ever be gotten under control by its users except in minor ways.

Tests

Before going further let me say a word about testing. I do not advocate the elimination of testing because it is very necessary but I wish to point out the fallacy of thinking that by testing we can determine the reliability of metal made by a process that breeds unreliability. Testing, both physical and chemical, can only show

the characteristics of a relatively small part of the mass because only a small part of the mass can be tested. As an example to bring out the point that I am trying to make, I will cite the case of the Mount Hope Bridge. This was a cable suspension bridge. Up to this time all of the steel wire used in bridge cables had been made by the acid open-hearth process and of completely drawn wire. The steel for the Mount Hope Bridge was made by the basic open-hearth process and only given a single draw through one die. The wire was then heat treated to get the required physical characteristics. It met all the required physical tests and was put into the cable and the bridge built, with the result that the bridge failed before completion. In the physical testing of wire for a bridge cable, we must remember that we test physically or chemically only the ends of each strand of wire and that we must depend for our reliability for the rest of the mass entirely on the process. Here where the more reliable acid open-hearth process was discarded for the basic open-hearth process and where the full drawn wire (which process tests the whole mass of the wire) was discarded for a single draw and heat treatment, dependence was placed on testing and the result was failure. I have cited this example because to me it is an outstanding one which proves how great a part the process plays in insuring reliability and how little dependence can be placed in testing.

And now I must discuss the subject of welding because it plays a great part in present day steel making. Many believe that it is proper to cast an ingot with blow holes or other voids and depend on the welding that takes place under the pressure of roll, hammer, or press to unite these disconnected surfaces. That this does not reliably take place is, I think, best proven by the prevalence of laminations, blisters and such similar flaws; by the abandonment of welded locomotive frames; by the fact that the efficiency of rolled welded pipe is only about 60 per cent for butt welds and 80 per cent for lap welds. Furthermore, pressure welds are only applicable to the soft, mild, weaker steels.

A tremendous amount of thought and effort has been put on the subject of welding, a national society has been founded to coordinate this thought and effort and wonderful strides have been made. Two new welding methods have been developed, namely, flow welding with the acetylene torch, and fusion welding with the electric arc. These new welding processes may be vastly superior to the old pressure welding and are having a marked effect on

industry. Their use may be eminently proper where solid metal cannot be used but I think that the advocates of welding in their enthusiasm tread on dangerous ground when they use welds where welds are not necessary. For example, welds are not necessary in the making of tubing because we have the Mannesmann seamless process. However, welds may be necessary in the joining together of the various lengths of tubes because we are limited in the lengths that we can transport. Irrespective of what proof may be advanced, I do not believe that a weld can ever be the equal of sound rolled or forged metal, for a weld at its best can be only a casting and castings, according to the teachings of metallurgy, are not equal to forgings and their use is proper only where strength is not a consideration or where the necessary shape makes forging impractical.

I am calling particular attention to tubing because it is a product of large tonnage and because at the present time the whole tube industry seems to be getting ready to change over from pressure welding to electrical fusion welding. This, while it may be a step in advance, is still only a change from an unreliable process to one still unreliable, though possibly less so, and I believe that if tube makers would give the same thought to the Mannesmann process they are now giving to fusion welding, it can be developed to where seamless tubing can be made just as cheaply as welded tubing and we would then have our tubing supply made by a reliable process instead of by a relatively unreliable one.

I believe that the fusion welding phase of tube making is just one of the many makeshift phases that steel-making is so full of and that its end will be in sight almost before it gets completely under way, and I am taking this opportunity of so publicly recording myself.

Take two large tonnage products, namely, rails and structural steels. One would think that it would be logical to apply sound metallurgy to the making of these two products, but such is not the case. Rails are almost universally made in the basic open-hearth under a highly oxidizing slag. The carbon is usually worked down low, say about 0.10 per cent for convenience, and then recarburized with hot iron either in the furnace or the ladle with manganese and silicon additions in the ladle, the former sometimes in the furnace. The metal is then poured into big end down molds. The ingot is stripped hot before solidification is complete. Can any human metallurgical ingenuity devise a more unreliable process than this? Small

wonder that rails fail, that volumes are written on the subject, that the transverse fissure is always with us and yet those who direct our railroads do not seem to realize that they are still permitting their rails to be made by processes that give no chance whatever for reliability, processes that must produce rails full of flaws and that rails with flaws are not safe to make harder and thereby get better wear. It is my belief that only the constant and careful track inspection now in force on our railroads keeps us from having an epidemic of rail failures amounting to a national scandal. Railroads have found by using reciprocating parts on their locomotives made of sound alloy steel, that the unbalanced weights could be cut down and track maintenance costs thereby largely reduced. It is my firm belief that sound rails, though they will cost more to begin with, will be the cheapest in the end for the same reasons.

There is only one steel-making practice that can be worse than the practice just described for rails and that is the practice so largely used in making structural steel of so called killing the steel in the mold with aluminum. There is a steel works saying that when a heat is not good enough for anything else, kill it with aluminum in the mold and put it into structural products. A heat of steel made thus is likely to have as many different characteristics as there are ingots in the heat, because no two ingots will be alike, except that all will be dirty, all will be unsound and if cast in big end down molds all will be laminated because of pipe both secondary and primary along the axis.

Millions of dollars have been spent on mills to roll the universal I-beam and yet these beams are in many cases, and I believe most all cases, made in big end down molds of highly oxidized steel killed in the mold with aluminum. They have no chance of being sound or reliable and, due to the laminations or near-lamination that must be along their axis, are little more than two channels placed back to back and partially welded together. Factors of safety where present day structural steel is used are higher than would be necessary if this steel were made sound by a reliable process. This would mean a tremendous saving in the weights of the structures.

Modern buildings are being carried to tremendous heights. This has gone so far that in certain cases I believe that alloy steel has been used or is planned to be used in the lower floors of some of these modern structures. It seems to me that as our present day

buildings are safe with our present day factors of safety, with our present day unsound and unreliable structural steel, that if engineers would insist that structural steel be made by a reliable process there would be enough weight saved to make our buildings safe without going to any such extreme as the use of alloys which are scarce and whose use should be conserved for where their need is more imperative.

Plates are also made as a rule either by rimming or killing in the mold with aluminum. The steel is then cast in big end down molds. Sometimes the ingots solidify in the mold before charging and sometimes not. The product of these practices has no chance to be anything but flawed and unreliable. It must be unnecessarily segregated, it must be laminated, and in fact it must be both. One case has come to my attention where nickel steel boiler plates were cast in big end down molds. They had no chance to be anything but laminated along the axis of the ingot and thereby unreliable. Here again as in structural steels engineers use factors of safety high enough to save the maker of this flawed unreliable steel from failures and penalties.

The trend of the time is toward better and more reliable steel, and steel makers are under tremendous pressure to accomplish this. Unfortunately many of them are too deeply rooted in the traditions of the past, these being the accumulations of makeshift practices referred to in the foregoing. They do not seem to realize that their losses, rejections, chipping costs, etc., are all the direct and inevitable results of the unreliable makeshift practices under which they are making steel and that these wastes will continue to be with them until these unreliable practices are eliminated. At present no one knows when a heat of steel starts whether or not it will finish fit for the purpose for which it was ordered, because this depends on the guesses of the men making the heat, and men, even the best of them, do not and cannot always guess right; only science can predict with certainty.

And now a word to the users of steel and you are largely represented in this gathering. You can have steel as good as you desire if you insist upon it. You will not get it however if you permit it to be made by an unreliable process or practice and then try to determine by any possible collections of tests whether it is reliable or not. The reliability will come from the process and not from anything you can do. If the process is unreliable even though your test shows

right, you can be assured that the unreliability is in the mass and know only that it did not happen to show up in the relatively small part represented by your tests.

I have been purposely critical of present-day steel making in the foregoing; not critical just for the sake of criticizing but critical of practices that I am sure have no longer any rightful place in American steel making. If we did not know by research that we could eliminate the losses and unreliability due to these makeshift practices this criticism would be idle and purposeless, but such is not the case. Research has told us and we have only to follow its teachings. What are these teachings? They are many and many more than we will be able to touch on in this paper for here I shall limit myself to the fundamental considerations, the foundation on which we must build our steel-making process if it is to give us reliability. With sound foundations the rest of the building will be easier; the foundation will be the hardest step to take and must come first, because unless the foundation is sound we will be forced later on in the process to makeshifts and we will not get reliability. This is the trouble with our present-day steel making, the foundation is not sound.

Fundamentals Essential to Soundness

What are the foundations of a reliable steel-making process? They are the fundamentals essential to soundness that were mentioned in the first part of this paper and these fundamentals I shall take up in order and discuss them and from them build up what the steel-making process must be if it is to give us reliability.

The first fundamental is: "That the bath of metal be thoroughly deoxidized before teeming so that no chemical reaction can take place during solidification." If we consider deoxidation to mean the reduction of all oxides, solid and gaseous and the elimination of these reaction products, this fundamental can stand as written and I shall consider in this paper that it means just this. How do our present-day steel-making processes measure up to this requirement. To measure up we must remember that deoxidation to be complete cannot take place under an oxidizing slag. (See Gillett's discussion of my paper of 1929.)

With this requirement in mind the crucible process measures up well except that its slag has 6 to 7 per cent iron oxide but as this slag is acid, the iron oxide in it can be reduced, though at present

this is not done, and it then would measure up almost perfectly for there are currents in the metal in a crucible that give the ideal condition for the elimination of the reaction products of deoxidation. The crucible process, however, due to its cost, is probably not available to us nor is it possible to thus get deoxidized metal in quantities sufficient for modern industry.

The Bessemer process, acid or basic, does not measure up at all for it permits of deoxidation only in the crudest possible way and that only in the ladle. To my mind it is not truly a steel-making process but only an oxidizing process and it is fast by its own limitations taking for itself that place in our steel making operations.

The acid open-hearth process measures up well where it can be used for its slag at the end of the oxidizing phase of melting can be deoxidized and thereby changed from oxidizing to non-oxidizing or possibly even reducing and in either of these conditions permits of complete deoxidation as the term is used in this paper. If this deoxidized slag condition is not brought about, the acid open-hearth process has little advantage over the basic open-hearth process, its slag for a given percentage of iron oxides in it being still oxidizing though less so and it is possible to get just as dirty and unreliable steel out of an acid open-hearth furnace as out of a basic open-hearth furnace. If properly operated, however, it measures up to our requirements for reliability and its real limitation is that it does not remove either phosphorus of sulphur and therefore can play only a small part in our steel making.

The acid arc-type electric furnace seems to me to give little metallurgical advantage over the acid open-hearth. I have had little experience with it and its use is apparently mainly in the production of steel for castings where the supply of good scrap is ample and the convenience of small heats is the controlling factor.

We must depend for our steel supply mainly on basic processes because we must be able in the process to eliminate both phosphorus and sulphur. There are three basic processes available and these are the basic electric induction furnace, the basic electric arc-type furnace and the basic open-hearth. When used with an oxidizing slag there is little or no difference in these three processes, but when we are through with the oxidizing phase and want to deoxidize, the induction furnace has distinct advantages over the arc-type electric and the open-hearth. Due to the induced currents that the process causes in the molten bath, when a reducing slag is put on the bath,

all parts of the bath are brought into direct contact with the slag and this continues until deoxidation is complete. It is practically automatic and therefore the most reliable melting process that is at our disposal. It is, however, very costly to install and operate, has to date only been used in small units and is probably for a long time to come only available in a commercial way for the making of tool steel and such similar high cost products.

The basic arc-type furnace and the basic open-hearth furnace must for many years to come produce the great part of our steel supply and our choice must be between these two. When one has a large volume of high grade scrap carrying valuable alloy elements there can be no choice because the arc furnace permits of melting down with a reducing slag and the saving of these alloys. Where phosphorus and sulphur must be removed from the charge I see little metallurgical difference in the processes. Both must use an oxidizing slag to eliminate phosphorus and any other element not wanted and having gotten these into the slag by making it highly oxidizing we cannot change this slag from oxidizing to non-oxidizing or reducing without causing phosphorus to revert.

As the two processes are operated today it is customary to take the first or oxidizing slag off the basic electric and then build a second slag of lime and carbon (CaC_2) which is reducing. With the basic open-hearth the heat is deoxidized with the oxidizing first slag still covering the bath. The slag is shaped up and thickened as far as is practical to cut down the diffusion of FeO from slag to metal bath but this diffusion can only be slowed up and not stopped and it is this that prevents the basic open-hearth from getting complete deoxidation and makes it a relatively unreliable process, where the quality produced is dependent on the successful guesses of the operators.

They should not have to guess. If the deoxidizing is carried out under a non-oxidizing slag they will not have to guess and if we use a non-oxidizing slag for the finishing or deoxidizing phase of the basic open-hearth I see little practical metallurgical difference between it and the basic arc-type furnace, with its reducing slag. This slag reduces FeO from the bath under it where it touches that bath. The trouble is that it does not touch all parts of the bath. As long as the bath has currents in it, due to chemical reaction, the slag functions, but as the slag functions the reactions die out and the slag then only deoxidizes the top layers of the bath and the time required to bring

all parts of the bath into contact with the slag is so great that as Dr. Gillett stated the bottom comes up. It is customary therefore to complete the deoxidation of basic electric heats by adding deoxidizers such as silicon and manganese to the bath and when this is done the process loses all metallurgical advantages over the basic open-hearth when a non-oxidizing finishing slag is used. The two processes then become metallurgically identical and as between the two we choose the basic open-hearth because its cost of operation is very much less.

I do not believe that it will cost any more to operate the basic open-hearth as a two slag process than as a one slag process. I pointed out many reasons for this in my paper read in 1929. I also do not think that it will mean the abandonment of present-day plants but only their modification. For example when the open-hearth is equipped with tilting furnaces, the two-slag processes can be applied without change. When the furnaces are all stationary a certain percentage of these furnaces must be changed to tilters—the melting down and oxidizing phases being carried out in the stationary furnaces and the deoxidizing and finishing in the tilters. I would today estimate that one furnace in five must tilt. There can be no possible doubt that where we are only going to melt down and oxidize in a furnace its operation can be made much more rapid than if we must have at the end of these phases a slag under which we can finish the bath. Probably when we tap from the oxidizing furnace we can in the ladle add deoxidizers and get rid of most of our oxides, putting this partly deoxidized metal into the finishing furnace in about the same conditions that the bath in an arc-type electric furnace is in when the deoxidizing additions are made in the furnace. The function of the finishing furnace then is only to complete deoxidization and hold the bath fluid while the flotation of the final reaction products of deoxidization take place. This non-oxidizing slag in the finishing furnace need not be lost with each heat, only enough being tapped out to properly insulate the metal in the ladle while it is being poured into the molds.

Our basic open-hearth furnace operated thus becomes a reliable process free from guesses, for chemically our bath does not change in this finishing furnace and we are in a position to have an accurate analysis of each heat before tapping. Should the bath after analysis be low in any element this can be added as can any alloy element wanted without fear of loss. This gives us a reliable process for the production of clean deoxidized molten metal in large volume and at

a cost in my opinion not greater than our present-day partly deoxidized and unreliable metal. Our next step in the process is to cause this metal to solidify free from shrinkage flaws.

A bath of deoxidized steel when poured into molds solidifies by dendritic crystallization and this makes necessary the second fundamental for sound steel which is "that the metal be cast in an ingot mold whose inside dimensions are enough greater at the top than at the bottom to prevent internal bridging." This has been proven so many times by so many men, among whom I may name Howe, Priestly, Gathmann and mathematically by Field, that I do not think it necessary to add to the volumes written. Already practically all of the truly sound steel made is cast in the big end up mold and it is one fundamental to soundness that can hardly be questioned by anyone. The cost of big end up molds is slightly greater than of big end down molds, the present makeshift standard, but this cost difference is much less than most people think and is a matter of a few cents per ton only. Where plants are properly equipped to handle big end up ingots there is no difference in the stripping costs.

This brings us to the third fundamental to soundness which is "that the ingot be held in substantially a vertical position until solidification is complete." The natural law that steel follows in solidifying is that dendrites or pine tree crystals form on the chilled surfaces and grow inwardly in the mother liquor. The rate at which these form and grow is directly proportional to the rate of chill or rate at which heat is taken from the molten mass. The number and size of these dendrites is also directly proportional to the rate of chill. This was excellently brought out by Priestly in a paper read some years ago before this Society.

After these dendrites have grown inwardly for a while the temperature of the molten metal in the core of the ingot has fallen to where it is near the solidification point and at this stage free or equiaxed crystals form all through it. The rate at which the metal cools governs the size of these crystals and if any attempt is made to strip before the ingot is completely solid the crystals will be unduly large and this result is called ingotism. These large crystals can be broken up later on by rolling and subsequent treatment but as the intergranular-nonmetallic material collects in well defined areas between these large crystals their bad effects can never be eliminated and the result is a structure commonly called woody and very weak against transverse stresses.

The things that we can do to eliminate, as far as possible, this unreliable condition are therefore to melt and deoxidize as completely as possible so that there will be a minimum of nonmetallics and then to solidify this metal as rapidly as possible. It is obvious that the ingot must stay vertical during this solidification.

The fourth fundamental to soundness is "that a refractory sink-head be used in all ingot molding." I said at the beginning of this paper that this was not truly fundamental to sound steel, that it was only commercially necessary to make steel of this character at not too great a cost. This I think needs explanation.

The function of a sink-head is two-fold. First it maintains a pool of molten metal to feed the shrinkage of the metal as it takes place. Secondly, it must maintain a pressure head on the metal in which the crystals are growing. The first function is quite generally understood. The second is frequently overlooked. Unless the pressure head is maintained the inwardly growing dendrites cause small local bridges between their lateral branches and these branches trap local areas among themselves which, unless these branches are bent back on themselves by pressure, leave small local voids among the dendrites and probably between the equiaxed crystals also.

In other words, the crystal does not have enough metal to complete its growth and therefore there are voids among the crystals, not necessarily only along the axis of the ingot but in many places throughout the mass. It is for these reasons that the putting of a sink-head on a standard big end down ingot does not produce sound steel. Bridging takes place and the whole central core of the ingot is flawed because in addition to secondary pipe the crystals in this area do not have enough metal to finish their growth. The result is that an axial area of flawed metal comprised of partially starved crystals equalling about 10 per cent or more of the total cross-sectional area of the ingot begins a short distance under the sink-head and goes practically to the bottom or at least down to the cleavage cone.

Up to the present time the cost of sink-heads has been a strong deterrent to their wide use. These costs have ranged between 60¢ and $1.00 per ton of ingot produced. This need no longer be such a drawback for the writer has recently seen operations where sink-heads were produced for 20¢ per ton of ingot with prospects that with a fuller use and a better chance for mass production their cost would drop to about 12¢ per ton of ingot. These figures and the

small extra cost of the big end up mold show that the two combined are not enough to be a serious drawback to their much wider use.

The biggest drawback is the temperature difference that the big end up ingot has at its top and bottom when the ingot has solidified. This difference must be brought about to prevent bridging and thereby get sound ingots and it takes considerably longer to heat this ingot than a standard big end down ingot which can go into the soaking pits at a much more uniform temperature. When the heating capacity has been worked out and built to supply the mill with hot ingots of the standard type, the use of big end up sink-head ingots slows down the operation and adds considerably to the cost. More soaking pits must be built. When this is done the extra cost is very much lessened and is brought down to a figure that puts it well within the bounds of present day commercial practicability for tonnage steel operations.

There are two other factors that enter into the reliability of steel and these are cleavage of dendrites and the re-oxidation that takes place during pouring. These we will discuss in order.

First Cleavage: Much research is needed on this subject for I do not know of satisfactory explanations of some of the damaging effects that we know are produced where this so called cleavage of dendrites occurs. My own observations are as follows: where dendrites grow side by side with their main stems approximately parallel with each other their branches seem to make a perfect union; where the main stems of the dendrites are not parallel the tips of the dendrites do not seem to form a true union. There is a marked area of weakness in the plane containing these tip junctures.

These cleavage planes are present in all ingots cast except the round ingot. They occur wherever there is a corner or salient the equivalent to a corner. If we could cast round ingots the answer would be simple but we cannot. In round ingots the mold expands away from the ingot leaving the ingot skin or shell unsupported and subject to the internal ferrostatic pressure of the molten mother metal which bursts this skin or shell thus causing bad vertical seams and streaks.

This lead to the corrugating of ingots where the mold expands inwardly and supports this skin or shell until danger of internal pressure rupture of the skin has passed. This corrugating however puts corners or salients into ingots and at these corners cleavage takes place. Furthermore, our whole system of rolling is based on passing

ingots between cylindrical rolls and these call for square or rectangular sections of ingots if proper and most efficient rolling conditions are to be maintained. With these we have the corner cleavages.

The most promising chance that we have of getting rid of the cleavage seems to be the creating of currents in the metal to break up and disperse this zone or plane in which the dendrite tips meet. This must be done mechanically. European practices have been reported where ingots are rotated during the early stages of solidification to accomplish this. I believe that a reciprocating motion would be better than rotation.

Second: Re-oxidation during tapping and pouring. We may deoxidize a bath of metal as completely as we desire if we do this under a non-oxidizing slag. To get this molten metal into ingot form, however, we with our present methods must pour it through air into the molds. Oxidation must take place. The total amount of oxidation that does take place if dispersed and diluted into the whole mass is probably not enough to cause unreliability. If, however, it is concentrated in spots it causes trouble.

We have two means of pouring, namely, top pouring and bottom pouring. In bottom pouring we undoubtedly get a better dispersion and mixture of this pouring oxidation. We get this, however, at a price that to my mind is prohibitive. First it costs $1.50 to $2.00 per ton of ingot and second we dirty our steel with the nonmetallics washed from the runner bricks and joints. There are other disadvantages which in many tonnage operations practically prohibit bottom pouring. Personally, I do not believe in it.

In top pouring we get a dispersion of the oxidized metal except where this metal splashes up against the side of the mold and here if it does not melt down it is likely to cause us trouble. These splashes in my opinion cause the thin skin of fine crystals that form the outer skin of ingots. Under these we have the zone of dendrites and inside the equiaxed crystals. This has been completely and well described by the British Ingot Committee in their report, and by Fry in his excellent paper on Locomotive Forgings, read before this body in 1929.

This thin skin may or may not be present or it may cover part of the ingot surface only. Wherever it appears it is always oxidized and full of small blow holes. These are undoubtedly a source of weakness and I believe the nuclei of many seams. I do not know how

to correct this but I consider it one of the most fruitful sources of chipping costs.

Mold washes are certainly not a remedy though they probably help. Pouring boxes help but they are certainly not the cure as the steel still has to drop 6 or 7 feet to the bottom of the mold. Possibly the answer lies in some practical development of a practice that was used many years ago in pouring armour-piercing shells. This was to use an extension tube below the nozzle, let this go down to the bottom of the mold and lower the mold down as it filled.

I have purposely called special attention to these two phases, cleavage and oxidized splash because to my mind we have as yet no real answer to either. Both ultimately show up in bad surface. Chipping costs in a well managed tonnage steel plant will total today close to 10 per cent of the total earnings of that plant. The indirect costs and losses due to bad surface will, I think, total as much more and this is much too great a cost load for the industry to stagger under.

No makeshifts, such as mold washes and pouring boxes will give us the answer. True research is badly needed. At present as far as I know none is being done and I am hoping that steel makers when they see the problem more clearly in its true light and realize its importance to them will get together and organize and find the proper answer to these two most puzzling phases of steel making.

Conclusion

And now in conclusion. I have tried in this paper to bring out what the foundations of a reliable steel-making process must be. The ingot is, of course, the end of the steel making stage and this must be sound, of clean metal and as free from lack of uniformity such as segregation, as natural, physical chemical laws permit.

From this point on all that can be done by rolling, shaping and subsequent treatments including heat treatment can only affect the crystal structure and nature and shape the mass for use. It can be spoiled but not improved as to soundness for this is determined when the ingot is made.

I have called particular attention to such makeshift practices as rimming steel and killing steel in the mold with aluminum because these by their very nature can never be reliable, can never be controlled processes and I think have outlived their usefulness. They should no longer be a part of our steel making.

The big end down mold was put into use in the early days of tonnage steel making when the metallurgy of solidification was not understood. It can produce no steel in ingot form that can make even any pretence at soundness and thereby reliability. It should no longer be used. Its makeshift substitute, the mold with no taper, is little, if any, better.

I have called particular attention to the fallacy of letting steel be made by unreliable practices and then trying to determine the reliability by testing. It cannot be done. If buyers want reliable steel they must at least specify the foundations of the process under which the metal must be made. They then have a chance of getting what they want otherwise they have none. What these foundations are and the reasons that they are the foundations is gone into as completely in this paper as space permits.

DISCUSSION

Written Discussion: By H. H. Ashdown, consulting metallurgist and engineer, Y. M. C. A. South Chicago.

It is a privilege for one to express admiration for the straight and open manner in which the author has presented his views. The paper is deserving of much careful thought and should result in material improvement in wide fields of manufacture.

The writer would suggest the author modifies, on the second page, his second and fourth clauses, to read "except under special conditions." Although the principle of casting ingots big-end-up is correct, ingots both large and small, the former in particular are cast in quantity small-end-up perfectly sound and solid. Success depends on taking sufficient care of the controlling conditions. With careless casting conditions it is not uncommon to find axial unsoundess in ingots cast big-end-up. For certain classes of work the small-end-up ingot offers marked advantages.

Regarding No. 4 a refractory-lined sink-head is not always to be desired. Unsound steel for certain purposes offers distinct advantages, as in the case of certain types of leaf springs. Again for ingots intended for certain classes of hollow forgings from which the core is trepanned, pipe is of little moment and the absence of nonmetallic inclusions from the refractory sink-head is of vital importance.

The general statements made on steel making will receive full endorsement of the progressive steel-works management, particularly regarding additions to the ladle and more so, however small, to the mold. It is impossible for the products of any reactions to become liberated from a viscous mass, even if the reactions were complete.

On the fourteenth page, the author referring to round ingots infers that if such could be made many ingot problems would be simplified but states this form of ingot cannot be cast. That statement is incorrect as will be seen on

page thirteen of the paper No. 30, now before this session where ingots up to 134,400 pounds (60 tons) weight are illustrated and examples can also be shown of groups of small ingots 1500 pounds produced in tonnage quantities for mill fabrication.

Referring to the subject of crystallization as outlined on the twelfth page, although by a normal process of solidification dendrites may form and grow to ascribed law, the statement that the rate at which these form and grow is directly proportional to the rate of chill or rate at which heat is taken from the mass appears incorrect. By an accelerated rate of cooling the formation of dendrites, as generally accepted, is entirely prevented and gives place to equiaxial crystals.

The formation of the fine crystals forming the outer casing of ingots appears to be the result of a function of temperature and rate of teeming and not the outcome of splash oxidation. They may also result from the quick freezing of the thin eddying waves caused by the fall of the liquid stream, washing against the mold walls. The thin upper relief wall of partly cast ingots is usually built up of these fine crystals.

The suggested use of an extension tube or trumpet suspended below the ladle nozzle to avoid splashing, is of questionable value. The wash action of the liquid steel on the refractory lining of the tube carries foreign matter into the ingot, and the writer on more than one occasion has seen the metal freeze in the tube, causing increased difficulties.

The author's reference on the fifteenth page to the rotation of the ingot mold during casting is of interest as it was my privilege to be in the plant in the English Works where this method was first inaugurated. This method offers many advantages, by directing the liquid stream toward the mold wall the dendritic structure is less pronounced, the cooling gradient less steep, contractional stresses reduced, the sonims are kept in the center and eventually brought into the head and the ingots generally are sound and clean.

Referring to a suggested reciprocating motion of the mold I would refer the author to Dr. Hultgren's paper before the British Iron and Steel Institute 1929 where he deals with this problem experimentally on 12-inch ingots. Although the dendrites are to some extent broken up and a larger proportion of equiaxial crystals produced considerable carbon segregation occurs and Dr. Hultgren in his own words says "it hardly gives promise of practical application." To apply this motion to large ingots one might anticipate, apart from other problems, considerable skin rupture due to ferrostatic pressure, as the ingot at an early stage loses support from the mold walls.

Written Discussion: By W. H. White, open-hearth superintendent, Duquesne Steel Foundry Co., Coraopolis, Pa.

Mr. Dornin's paper, if I read it correctly, calls for more scientific methods of making steel. It seems he has failed to see any progress in this respect in the past ten years or more. It may be true that this progress is limited but it has and is ever being made.

The American Society for Steel Treating in dealing with this subject would naturally make slower progress than if they were in their own direct field, heat treating. The way to make improvement in any line of production

is not always under scientific methods, the education of the individual actually doing the work means much in this respect. To educate and train a steel maker requires another steel maker who has the knowledge through years of successful experience. It is foolish to think we are riding on railroad rails today of no better quality than ten years ago, for the makers of rails have improved their steel, making wide and intelligent research even though they have not seen fit to convert a basic open-hearth into a two-slag electric method of production.

The cleanest and strongest piece of steel made may break on a zero morning and the natural elements play their part in this direction. The research work that some men have made, has taught them more than they were able to give in many cases, and it is regretable that so many intelligent men who would be able to contribute progress to steel making have found it uncomfortable to even stand in front of a furnace during a complete heat making, much less use a shovel, rabble or test spoon.

I will admit that the average melter lacks much metallurgical knowledge but in its place he has the greatest of all teachers—Experience, and if he is a thinker, he will make progress and reliable progress also.

Testing of the right character is reliable and not only tells us whether our steel is made right or not, but is the answer to whether it has been treated right or not. If the method of testing has not been properly recommended, that is not the fault of the maker.

If you look back over the few years that this subject has been before the Society, the most knowledge has come from the men who actually have had the responsibility of making steel and they are few, because but few can or care to put it in writing.

Good slags, good steel, proper furnace operation, is not guess work as stated, but real judgment manipulated as conditions confront the maker in the ever-changing metallurgical reactions that occur on any heat of steel.

Give the untrained man a scientifically controlled furnace or method, and he will still produce bad steel for you. Improve the personnel as you improve the method and progress reliability will be made.

Mr. Dornin is to be complimented on his paper, but I do feel that steel making is still much of an art executed only as efficiently as the skill of the individual will allow. To make improvement, the individual must be improved as well as the method.

Written Discussion: By T. McLean Jasper, research department, A. O. Smith Corp., Milwaukee.

If this paper can only awaken the makers of tonnage steel to realize the tremendous amount of trouble that steel using industries encounter by the introduction of unreliable steel into their processes, it can be of very great value.

The writer agrees with the author that progress for improvement in tonnage steel making has been unusually backward in general. The demands that industry has put upon the steel maker has been strenuously resisted by many of the larger steel mills. There is such a variation in the manner in which steel from various heats will go through a manufacturing process that, to the steel user, it is very evident that the tonnage steel maker does not have a

satisfactory control of his product. The ordinary laboratory tests do not allow us to select the best steel from steel of questionable value.

With uniformly clean sound steel the fabricator of steel products could do so much more for his customer. The economics of the utilization of steel should have for its purpose, longer life or greater resistance to corrosion, greater strength and energy resisting qualities, and greater uniformity of product.

I disagree with the author that welding need be unreliable, yet I believe that in many cases it is a makeshift in the hands of many welding operators. I believe that for those who are conscientiously developing and improving this process, there is a possibility of developing the full strength, ductility and energy resisting qualities of the steel joined without the appearance of any zone of weakness adjacent to the weld. It is from the demonstrated fact of the improved qualities of the deposited weld metal over that of the steel in the plates joined and over that of the steel in the weld rod used, when proper welding is performed, that the writer makes this observation. These facts also lead to suggestions of ways and means for improvement in steel making.

The nonchalant, and may I say careless, attitude of many good men in calling a weld a casting, in the sense that they wish to convey the idea that the casting need be full of blow holes and have other very objectionable qualities, may be caused by their association with careless foundry practice, when using steel for the castings which has been made by the so called makeshift processes, and by their association with careless welding. These conditions need not prevail as is well demonstrated by the results obtained from consistently sound, strong and ductile steel castings and from consistently sound, strong and ductile welds. Much has been done by the earnest metallurgist in finding ways and means of overcoming steel casting and welding difficulties.

I agree sincerely with the author that there is a mass of reliable metallurgical data which if applied to tonnage steel production would cause a vast improvement in the general quality of steel offered on the market. This improvement would produce a large economic saving to the final user of products made from such steel and result in great economic advantages to the country at large.

I admire the author of this paper for the blunt honest manner in which he states his beliefs. I agree or disagree with him sincerely and wish to state that papers of this kind, if taken seriously, and honestly analyzed, can have but a good effect upon the steel industry at large.

Written Discussion: By W. J. Priestley, vice president, Electro Metallurgical Sales Corp., New York City.

I appreciated the courtesy of your President in being asked to discuss Mr. George Dornin's paper on "Process Reliability in Steel Making." I am sure this paper contains a number of new thoughts which will be of interest to steel makers in general.

Personally I think the status of open-hearth steel making is not as bad as the author states. Investments are made in the steel industry for profit and there are instances where it would not be economical for the consumer to use

the highest grade of steel produced at a higher cost in places where the need might be met by using steel which could be produced at a lower cost.

In reference to the author's comments on rimmed or effervescing steel, I think there are places in the industry where this type of steel will meet commercial demands better than a thoroughly deoxidized steel. A well made rimmed or effervescing steel may be made free from dirt and nonmetallic inclusions and this type of steel has a specific use in the steel industry.

Were it not for the item of cost, it would be desirable if all steel might be made perfectly, but since steel companies are in business for a profit, there are instances where weight and mass are essential things and for the consumer to use the finest quality of steel at an increased cost would be superfluous. On the other hand, where greater strength and higher physical properties are required, combined with less weight, it is desirable for the consumer to use higher grade alloy steels even though the unit cost is greater.

I believe the knowledge of steel making has made noticeable progress in the past ten years. Due to more rigid specifications and inspection, closer supervision has been given to the making of steel. Competition has also done much to stimulate the quality and improve methods of making steel, and certain economical factors have contributed to the improvement in the manufacture of steel. Oil and natural gas at one time were looked upon as luxuries in melting open-hearth steel. Due to higher labor costs, fuels of this kind have in many cases turned out to be more economical than producer gas used in former times when each plant had their own battery of gas producers. The introduction of oil has speeded up the time of melting, given better metallurgical control and in other ways has become more desirable than former methods of heating.

Real benefits have come from papers and discussions carried on by technical societies towards advancing the standards of steel making. Everyone is striving to do his job better and it has been possible to obtain valuable information by exchange of ideas which were not formerly available. Improvements in furnace design, equipment and refractories have also aided in the progress of steel making. Certain companies supplying raw materials and other supplies, have established service departments composed of expert technical men who have done much to stimulate interest in making better steel. Our own company has a staff of practical steel makers and a large research laboratory for broadening the use of special alloy steels and helping steel companies who have limited laboratory facilities in solving special metallurgical problems.

For a number of years, we have been advocating the use of manganese in the form of spiegel and 15 per cent ferrosilicon pig as desirable agents for cleaning up open-hearth steel in the furnace. The elements manganese and silicon are both strong deoxidizers and the combination of the two forms a manganese silicate slag which has a low melting point and readily rises from the bath of molten steel into the slag. We have not recommended the use of 15 per cent ferrosilicon alone since the product of oxidation forms a silicate which is inclined to be refractory and will not coagulate and rise from the steel as freely as when it is combined with manganese oxide in the form of a manganese silicate slag.

To meet the demand for cleaner open-hearth steel, we have introduced a new alloy containing 7 to 8 per cent silicon and 25 to 30 per cent manganese which combines in a single alloy the desirable elements manganese and silicon. The alloy contains these elements in the proper proportions to give the best results. Silico-spiegel is now on the market and steel treated with this alloy has proven very satisfactory in making clean basic open-hearth steel.

I was particularly interested in that part of the paper dealing with duplexing from a stationary open-hearth through a tilting open-hearth furnace. Since I have had no experience in making steel by this process, I am not in position to discuss it but I thought the readers of this paper might be interested in a review of facts and figures gained by duplexing steel from a stationary open-hearth through an electric furnace while making high quality steel for ordnance material subject to transverse tests. If duplexing is desired as a means of making higher quality steel, I believe the basic electric furnace offers many advantages for refining and deoxidizing which can not be obtained in a secondary open-hearth furnace.

In transferring molten steel from the melting down open-hearth furnace to the refining and deoxidizing furnace, there is a considerable drop in the temperature of the metal and under certain conditions, it will freeze around the hearth line. The time required to re-heat a bath of steel in the deoxidizing furnace may be done quicker in an electric arc furnace due to the greater heat input in a given period of time. Also the refining slag may be melted and shaped up more satisfactorily in the non-oxidizing atmosphere of an enclosed electric furnace than in an open-hearth where air must be added with the fuel to support combustion.

While it might be possible to obtain in a secondary open-hearth furnace a slag low in iron oxides, through the use of fresh additions of burnt lime, there would be no positive proof that the steel was free from oxides. Instances have been seen where a strong carbide slag, apparently well deoxidized in a basic electric furnace, has been re-oxidized and turned back to the brown state by the simple operating of rabbling the bath of steel. This cycle of re-conditioning the slag and re-rabbling has been repeated four and five times before the bath of molten steel approached the degree of deoxidation where the slag would no longer change color after rabbling.

It may be of interest to furnish a typical average analysis of slags taken from a basic electric furnace where the charge was melted in a basic open-hearth furnace and refined in a basic electric arc furnace.

Typical Analysis of refined Slag (Per Cent)

SiO_2	Fe	Al_2O_3	Mn	CaO	MgO	P	S	CaC_2
10	0.63	2.50	0.40	60	14	0.046	0.30	1.21

A typical analysis of steel made under the slag mentioned above was as follows:

C	Mn	Si	Ni	P	S
0.30	0.70	0.22	3.00	0.013	0.008

The interesting thing about this analysis is the low sulphur which is indicative of the degree to which the steel was deoxidized. The refining operation which produced the low sulphur also deoxidized and cleaned the steel of non-metallic impurities.

The author mentions the layout of one tilting open-hearth furnace for refining to supplement five stationary open-hearth furnaces used for melting down. An electric furnace with greater power input would accomplish this refining in one-half the time and, consequently, would take care of twice the number of open-hearth furnaces used for melting down.

There are many factors which make the operation of an electric arc furnace cheaper today than in the past. The cost of electricity and electrodes, which are the two principal items of expense in the operation of an electric furnace, are lower today than they have been in the past. This is due to the lower price of electric current and to the improvements that have been made in both graphite and carbon electrodes with respect to quality and sizes available for use in electric furnace melting. Graphite electrodes are now made commercially up to 18 inches in diameter and amorphous carbon electrodes up to 40 inches in diameter. These sizes provide for a larger power input which permits the use of larger furnaces, speeds up the time of melting and eliminates other items of expense which formely worked against the manufacture of electric steel.

With the new conditions mentioned above, it seems apparent that anyone considering a duplex layout as a means of obtaining higher quality steel would install an electric arc furnace instead of a tilting open-hearth furnace. Anyone considering large units for the manufacture of high grade steel on a large scale basis might be interested in considering the electric furnace for both melting down a cold charge and refining in the same furnace. With sufficient power and large sized electrodes now available, a charge of cold scrap may be melted down at a rate fairly comparable to the time required in an open-hearth furnace using gas or oil as fuel.

With the facilities for slagging off, deoxidizing and refining afforded by the use of a tilting electric arc furnace, it therefore seems desirable to use this type for finishing high grade steel regardless of the type of furnace in which the initial charge is melted, provided it has been decided to manufacture duplex steel and an electric furnace is available.

Written Discussion: By J. M. Gaines, Jr. Cambridge, Mass.

With the general trend of Mr. Dornin's observations most people will agree unreservedly. For it is certainly a time when the steel maker needs to consult the researcher, with a view to improving the quality of his product. Many are already doing so. Certainly, too, there are in existence many antiquated practices, which the author, in timely fashion, drags under the spotlight of his criticism.

One wonders, however, at the assurance with which it is stated that steel must be completely deoxidized before teeming, and all the inclusions thus formed eliminated. The question naturally arises as to whether a steel with one one thousandth of a per cent of silicate or alumina particles may not be just as reliable from the standpoint of service as one which contains no detectable non-metallic constituent. It is, I believe, still a more or less open question as to whether or not a certain percentage of the proper kind of inclusions may not actually be desirable. In one definite instance[1] the practice of dead-killing with

[1]Abnormality in Case-Carburized Steels. Co-operative Bulletin 45, Mining and Metallurgical Advisory Boards. Pittsburgh, Pa., 1930.

silicon or aluminum has been shown to lead to abnormal structures in case-carburized steel—and this, not because of the presence of inclusions, but of the unoxidized deoxidizer. While it is granted that only a small tonnage of basic open-hearth steel is carburized, the example is still valid as an instance of information gained from research on one phase of a problem, and its relation to an entirely separate phase.

Evidently what Mr. Dornin wants in a process is essentially the ability to control the reactions, and on that we are agreed. One of the biggest tasks ahead of the research worker is the development of suitable indicators. In the absence of such indicators, Mr. Dornin proposes to insert, in the usual basic open-hearth process, a stage which will take place so slowly that there will be time to make a complete analysis of the heat while the inclusions formed by deoxidation are being floated out under a neutral slag.

Under proper conditions—i. e., high temperature, agitation, and the presence of oxides of iron or manganese for fluxing—inclusions can be eliminated fairly rapidly and completely. If, however, enough deoxidizer is added to remove all the reducible oxide in the bath, there will be no agitation, no fluxing, and the particles will be of a uniformly small size. Under these conditions the rate of elimination will be painfully slow, and it is doubtful that the goal of absolute cleanliness—or even of satisfactory cleanliness—can be reached in a length of time not prohibitively expensive.

Since the subject of modification of the process has been brought up, an alternative suggestion may be in order. There is a certain amount of evidence[2] to show that steel at the end of the oxidizing period contains more FeO than satisfies equilibrium conditions with respect to the carbon content. There is thus a condition of supersaturation which may be relieved by the introduction of solid bodies into the steel bath, causing reaction to proceed with the evolution of CO as a gas. Such is the familiar action known as "boiling on a rod." Under a neutral slag, a large part of the deoxidation could be carried out by rodding, which is, in effect, making the steel "rim" in the furnace. It is conceivable that seventy or eighty per cent of the FeO and MnO could be reduced in this way without the addition of any inclusion-forming deoxidizers. A small addition could then be made to "clean up" and the heat could be tapped at once in a clean quiet state.

The same line of action might be applied to the much-maligned Bessemer process. If, following the usual blow, a short blow of nitrogen gas be used, the unreactive bubbles, sweeping through the steel, will relieve the supersaturation by scrubbing out CO gas and will thus deoxidize the bath. The extra amount of nitrogen absorbed in this may would doubtless be negligible.

Written Discussion: By H. P. Evans, metallurgist, Pettibone-Milliken Co., Chicago.

Mr. Dornin has brought to our attention the inherent weaknesses of some of our steel-making processes in a very clear and concise manner.

I have been very interested in following Mr. Dornin's rational endeavors toward increasing the reliability of our present day steel-making processes during the past few years.

[2]See Round Table Discussion, Physical Chemistry of Steel Making, American Institute of Mining and Metallurgical Engineers, New York, Feb., 1929.

Steel producers all realize the fact that there is a large field for improvement in our open-hearth melting and that, with the rapidly increasing demands for a so-called high grade steel, even in our large tonnage requirements, some changes in our present melting and pouring practice will be necessary.

He has attacked the problem from the field of basic metallurgy and has laid down a group of fundamentals which are unquestionable.

I sincerely hope that the discussion which follows, will develop further ideas which will increase the practicability of adapting these fundamentals to our steel making processes and that any criticisms offered will be of a constructive type pointing toward better made steel.

Written Discussion: By C. H. Herty, Jr., supervising chemist, metallurgical section, U. S. Bureau of Mines, Pittsburgh.

Is is to be regretted that Mr. Dornin's paper devoted only two pages of 16 to the mechanical and metallurgical features of methods of preventing unreliability in basic open-hearth steel. Any steel making process, be it new or some modification of that existing today, will bear a most careful scrutiny, for whether we like it or not some of the present-day practices will be out of date before many years have passed. There is nothing more fascinating to the theoretical metallurgist than speculation on the possibilities of slags and their control. We must remember, however, that there is such a thing as diffusion and that oxidation of the metal in a basic open-hearth furnace may be slowed down tremendously by properly regulating the viscosity, temperature, and composition of the slag. For example, if a bath is dead-killed with silicon, FeO will be transferred from slag to metal at about one-fiftieth to one-hundredth as fast as when the bath is active. The ideal is, of course, no oxidation whatsoever, but this may not be as advisable as slow oxidation at a much lower cost.

To go to one or two specific points brought up in the paper: On the seventh page there are two statements which should certainly be commented upon: The first is that when a heat starts no one knows whether it will finish fit for the purpose for which it was ordered. It seems to me that this statement is as uncalled for as any I have ever seen published. I am of the opinion that the intelligent open-hearth operator has many of the phases of steel making too well in hand to be called an outright "guesser." Not that there is not much to be learned, for there is as much to be learned here as in any industry in the country. The second statement on the seventh page is that the users of steel "can have steel as good as you desire if you insist upon it." If I should go to a steel producer and say "You can have ingots without butt cracks if you insist upon it," I would probably also suggest that the particular mold would cost a little more than the one he was using. The same situation applies here. The phrase "and if you will pay for it" might well be added to the author's statement above. Not that the extra cost would necessarily be prohibitive or impossible to reduce eventually.

The author's discussion of crystallization and reoxidation is very interesting, and it is along these lines that many plants are working at the present time.

I hestitate to discuss the sections on rimming steel and welding, as my words would probably be only a flake in the snow storm. And if I should begin on the general subject of deoxidation as applied to the two-stage process and

to other points raised in the paper, I am afraid I would, as Andy would say, "be 'reliable' to go on forever."

W. J. REAGAN:[1] I might say that in regard to forging steel we average 90 per cent yield on our ingots. Our average rejections are 2½ per cent or under. We certainly do not start to make a heat of steel with any guesswork about it. Our percentage of rejections of entire heats is almost negligible. I certainly cannot agree with a lot of the conclusions. I think we have lots to learn yet from the open-hearth furnace, but I think it is only going to be a short time when iron oxide control is going to be a fixture in the basic open-hearth.

O. E. HARDER:[2] I am primarily interested in asking Mr. Dornin a question. I am concerned to know if he has found any relation between rimming steel, killed steel, the so-called better steels, made by certain processes and the phenomona of temper brittleness or embrittlement in galvanizing.

JEROME STRAUSS:[3] I would like to say first one word in favor of Mr. Dornin's remarks, even though it should be contrary to what the chairman has mentioned. There is more than one plant within a radius of 20 miles of the chairman's office in which the open-hearth practice is such that it is not determined whether a given melt of steel shall finish as 0.10 per cent carbon steel or a 0.50 per cent carbon steel before half of the life of that heat is over, in the molten state. I am not saying that to in any way disparage open-hearth operators, but only to remind us that the best practices that we know of today are not being followed by all of our steel makers. They still seem to feel, whether rightly or wrongly I will not say, that it is economical to continue with certain of these practices. On one other point I would like to take issue with Mr. Dornin. He makes a statement that leads one to believe that under no circumstances are castings equal to forged material. There are parts that are made in which stresses are in certain confined directions, and other parts in which stresses are applied in many directions. In the present state of the art forgings are anything but isotropic, and yet using the best steel making practice which Mr. Dornin advocates and applying to the solidification conditions of the castings the same care that he desires to apply to solidification of his ingots for the production of forgings, it is quite possible to produce a casting which is a very highly desirable product and which, bear in mind, can often be made quite isotropic.

Author's Closure

I have had no special experience with temper brittleness in galvanizing, but I have seen two angles made from the same heat that were galvanized and broken after they were punched. From one a fracture was perfectly tough and the other was evidently brittle. That steel was made by killing in the mold with aluminum. I do not think when you kill steel in the mold with aluminum you have a heat of steel. You have as many different heats as you have ingots in the heat. When you first start killing you do not know how much aluminum

[1]Assistant to superintendent, open-hearth department, Edgewater Steel Co., Oakmont, Pa.

[2]Assistant director, Battelle Memorial Institute, Columbus, Ohio.

[3]Chief research engineer, Vanadium Corporation of America, Bridgeville, Pa.

you will need. You put a certain amount in the first mold. You guess at it. For the second ingot you guess a little better. By the third or fourth ingot you think you have guessed right. As you go along in the pouring if you have a long heat the iron oxide from the slag to a certain extent changes the oxide condition in the steel, and you may have to change the amount of aluminum at the end. I think there is too much variation to call it under control. That is one of the things I term guesswork, not done willingly by the men in the industry, but because the process forces it on them.

There are just a few points I want to refer to. I will reply in full to the written discussion in my closing discussion. With regard to Mr. Gaines' suggestion as to how to get rid of most of the iron oxide before you make any additions, it seems to me that there is an endothermic reaction here and the loss of heat largely militates against what he wants to do.

Dr. Herty says that I only devote two pages to making basic open-hearth steel sound. It seems to me that I devote pages 8 to 17 to that purpose. The scientific data that convince me that too much of the open-hearth process has to be guessed at come very largly from Dr. Herty's own papers in scientific researches. One example is the fact that you have a deoxidation curve which falls to a point and begins to go up again as long as you have an oxidizing slag. You have to guess at that point.

To Mr. Ashdown, I do not think I can compromise on the fundamentals of sound steel making, which are that the reactions must be finished before you begin solidification, you must let solidification complete itself before you start reheating and you must use a big-end-up mold.

The cores illustrated by Mr. Ashdown seem to prove that these ingots cannot be solid. As I read the illustration in Mr. Ashdown's paper those cores are not sound. Round ingots, in my experience, can only be cast up to a limited diameter. I had considerable experience 30 years ago with round ingots, forging ingots, the fluid compressed ingots, and we could not cast them. The formula for a thin cylinder, pressure times diameter, equals the stress in the metal times the thickness of the metal militates against us.

In reply to Mr. Priestley, cost is the reason I think our present day methods are not adequate. There are the cheap, or so-called cheap, tonnage methods. They even look cheap. However, in my experience, they are not cheap in the end. They exact their penalties. The method described by Mr. Priestley, however, is too costly and too slow to be of any wide application.

In January when I am asked to read a paper before this session I always feel that I have something worth while to say and cheerfully agree. In May and June when the paper is due to be delivered to the Committee for preprinting it is, as a rule, not completed and I am always sorry that I have promised to write it. After the paper is preprinted and ready for presentation I invariably feel that I have not done the subject justice and always present the paper in the hope that through the discussion and my privilege of replying to that discussion I can in the end make the paper worth while.

Within the past year I have been in a steel plant going over the chipping records with the superintendent. The record shows heats of steel made for the same product which chipped at the rate of 20 tons per man per day and also

heats which chipped at the rate of one ton per man per day, the cost per ton in the first case being 50¢ and in the second case $10.00. These are rather extreme cases but cost differences ranging from $1.00 to $5.00 are common. This plant is that of a thoroughly successful company, its management is good and intelligent and its products bear a wide reputation. It gave the same care and attention to the two heats in question. The earnings of this plant will be roughtly $5.00 to $7.00 per ton of steel produced. It can easily be seen from the foregoing that something is wrong when two heats of steel, one following the other, are made by the same men and with the same care, one of which earns its full profit for the company and the other not only earns no profit but loses enough to kill the profit of the previous heat. The men operating this organization do not do this carelessly or deliberately. They try their best at all times to get uniformity in their product.

My belief is that the trouble in this case is in the steel-making process; that it is a process that the operators today do not control and it is also my belief that the process under which the steel referred to was made is not a process than can be controlled. The practice referred to is to so-called "kill the steel in the mold with aluminum" method. They tried rimming this steel and it is a grade of steel susceptible of rimming, for the carbon is between 0.10 and 0.15 per cent. They tried partly deoxidizing in the ladle with silicon and aluminum and adding only part of the aluminum in the mold. This latter combination seems to give them the best results. The steel is all top poured in standard big end down molds. The yield, ingot to bloom, is about 78 per cent after chipping bed rejections are taken out and this varies from as low as 70 to as high as 84. This picture is not an isolated case, it is typical of conditions that occur in plant after plant throughout the country. I have seen it occur in plant after plant for the past 15 years and it is my belief that it is little if any better today than it was 15 years ago. This is the side that interests the maker of steel. There is, however, another very vital side and that is the quality of the metal produced as it affects the user of steel. The variations here, as far as I can check them up, are quite as great as those that affect the steel maker. I believe from what my experience has taught me that the trouble lies mainly in the processes or practices under which most of our steel today is made.

Probably about 10 per cent of our steel production is fully deoxidized, cast in big end up molds with sink-heads and allowed to solidify before reheating. The remaining 90 per cent is all cast in standard big end down molds, some allowed to solidify before reheating and some not, some cast as rimmed steel in its oxidized condition and the remainder cast as partly deoxidized steel varying only in the degree of partial deoxidation. None of the practices just mentioned covering the production of the 90 per cent of our steel are in my opinion practices which give the operators control of the process nor can they do so. All are practices which produce ingots with shrinkage voids and depend on welding these during rolling, and the prevalence of blisters, laminations and seams, I think proves this expectation unattainable.

For these reasons I discussed welding in the paper as I did and also because it seemed to me that fusion welding of tubing was holding back and interfering with the proper development of the Seamless Process. Since writing the paper

I have been privileged to see pipe made by resistance welding as carried out under the Gustav Johnson patents and as described in the September number of *Metal Progress*. This process fuses two unoxidized edges together at or above the welding temperature and rolls the weld so that it meets the fundamental objection that I made to welded pipe in the paper. I do not know how reliable the process is and will leave its advocacy to its proponents, but in view of my later knowledge think that fairness requires this statement.

Referring to the 10 per cent of our steel made of completely deoxidized steel cast in big end up molds with sink-heads and allowed to solidify before reheating, I believe that this type of process is the only type that can be controlled by the operators. When these operators try to carry out this process at present they are handicapped by the oxidizing slag of the basic open-hearth and by the fact that our present day plants are mostly built and organized to make the unreliable partly deoxidized steels. The costs therefore mount out of all proportion to the results obtained.

I think our steel-making operating men are entitled to a sounder steel-making process than they today have to work with and this must come from our metallurgists. I believe that this body is composed of the best metallurgical minds in the country and that if help is to come it must be from here, and I now place the problem where I think it fairly beiongs.

I wrote this paper to emphasize how small a part of our present day steel production follows in its making sound metallurgical principles. Mr. Jasper in his discussion realizes this condition fully and I advise all makers of tonnage steel to read his discussion carefully. The fact that he and I differ as to the probability of getting a reliably sound weld by the electrical fusion method is relatively unimportant. He represents one of the largest users of steel in the world and his experience with the quality of metal obtainable under our present day tonnage methods is exactly what I would expect it to be, knowing the process under which it is produced as I do.

I think that I have already replied sufficiently to the other written discussions in my previous remarks. I wish, however, to say to Mr. Strauss that if steel is made sound and clean there is little difference between transverse and longitudinal tests and that the fact that a casting may be made isotropic would give it little if any advantage over a forging from this point of view and that it would still carry the fundamental disadvantages of a casting.

The whole aim and object in writing this paper has been to give to steel making operating men a process cheap enough to apply and which would give them a control of the quality produced which they do not now have and if it in any way forwards this object, it will have served its purposes.

STEEL INGOTS

By Herbert H. Ashdown

Abstract

The object of this paper is to show many of the difficulties met with in the manufacture of heavy ingots and to prove by both small and large scale experiments the cause of failure.

To show by applying the information obtained during experiment that these difficulties can be overcome.

To bring to notice the advantages to be derived by rotating ingots during the process of casting, and by rapid solidification arresting segregation, producing steel of more uniform analysis, preventing structural weakness, shortening the pipe and producing a larger percentage of ingot body of dependable quality.

THE reports issued by the Committee on "Heterogeneity On Steel Ingots" of the British Iron and Steel Institute in 1926 and 1928 have induced the writer to present this contribution which may add further useful information to this absorbing subject.

The early part of this data was prepared in 1914-16 and apology is offered for the crudeness of the illustrations as the information then sought was purely for works purposes and without thought of subsequent publication. The work was carried through at the expense of the Muroran Steel Works, Japan, and in view of the high costs involved in surface machining these large ingot sections, this was dispensed with as it was then considered the information required was obtained.

The illustrations represent a very small fraction of the large number of pieces similarly examined and it will be appreciated, they have been taken from the upper discard portion and trepanned cores of ingots, varying in weight from 179,200 to 268,800 pounds. This examination gives a ready index of the suitability or otherwise of the ingots for the purpose intended.

The photographs illustrating the heavily segregated areas are now common to us, particularly since the publication of the above

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Fig. 1—Half Lineal Section of the Upper Discard Portion of an Ingot.



Fig. 2—Segregates Diverted from Normal Upward Course by a Portion of the Crust.

mentioned reports. Perhaps of more particular interest is Fig. 1, which represents in half lineal section the upper discard portion of an ingot. The lower portion of this discard was cast in the "chill mold" while the upper portion was cast in a refractory-lined extension. This readily illustrates the effect of relatively quick and slow cooling on the location and volume of the segregates. Attention may be called further to Fig. 2, which shows the segregates being diverted from their normal upward course by a portion of the "crust" or "sonims" which form on the surface of large ingots during casting, and having been entrapped in the liquid steel. This

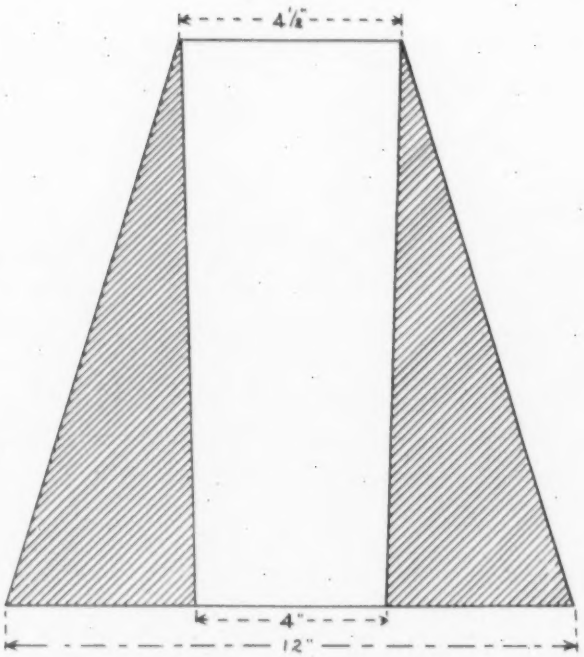


Fig. 3—Special Test Ingot Mold.

clearly shows the early stage at which segregation begins to form into mass areas, and having so formed, its tenacious character, and once formed, indifference to its passage through the more liquid steel.

The writer's interest in this subjcet has extended over a period of some 25 years, first when experimenting with 2000-pound ingots by applying lateral pressure to the walls of split ingot molds during solidification, with a view to liquating the segregate and preventing the formation of pipe.

At this same period ingots of similar weight were cast in molds placed on a slowly rotating table mainly with the same ultimate object.

With a view to illucidating some of the problems associated in casting heavy ingots, a number of small experimental ingot molds were made in direct proportion to the 268,800-pound mold. From the same ladle from which these heavy ingots were cast, steel was cast into the experimental molds and efforts were made to cool these down at the same relative rates as the full size ingots. These experimental ingots were sectioned and examined and it was found that the tendency to segregation was the same in these as in the full size ingots.



Fig. 4—Ingot Cast with Thick Section at the Base.

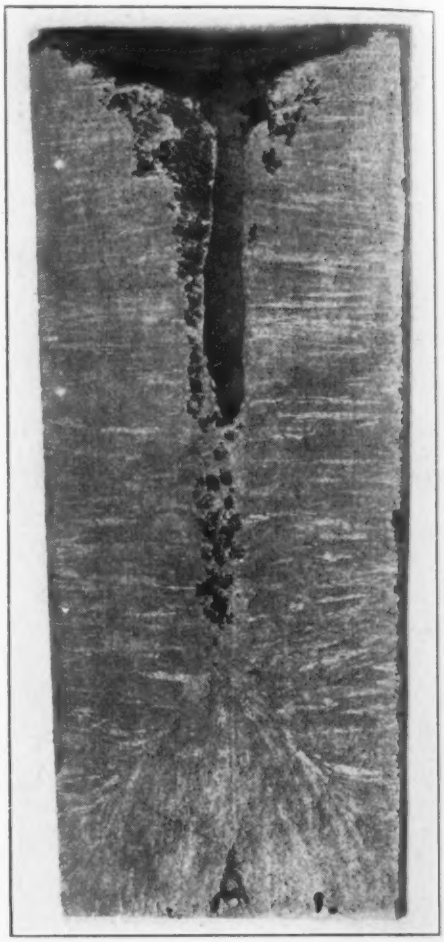


Fig. 5—Ingot Cast with Thick Section at the Top.

In these same experimental molds it was decided to cast zinc ingots, as in view of the characteristic properties of this metal to produce large crystalline structure, it was considered, from these, more helpful information might be obtained than that indicated by the use of sterine or wax. These ingots were caused to cool down under widely different conditions and the crystalline growth responded very successfully to the treatment given.

In the first instance, to study the chill effect of the mold walls on the metal, a mold although normally proportioned inside, was cast with a taper wall as shown in Fig. 3. Ingot A, (Fig. 4) was cast with the thick section at the base, while for ingot B, (Fig. 5) the mold wall section was reversed, the thick section being at the top.



Fig. 6—Ingot Cast in Normal Mold. The Metal was Cast Hot with Extreme Cooling.



Fig. 7—Zinc Ingot Cast at Normal Temperature and a Ring, Water Spray, was Applied from Middle-half Downward. Upper Half Kept Hot with Gas Ring.

The quicker cooling of the lower portion of ingot A, due to the thickened wall, not only illustrates its advantage in the production of a sound ingot, but also shows how the formation of the base cone is depressed and which, in steel, would be removed by a small percentage of the bottom discard.

Conversely, in ingot B, with a slow cooling base and quickly solidifying head, an extensive pipe is formed in addition to a long base cone, a portion of which would remain in the finished forging. As stated by Brearley, (Iron and Steel Institute, Vol. 11, 1917) in his paper on steel ingots, this base cone is anything but desirable, as a line of considerable weakness is developed, due to the junction of the chill crystals and also the enclosed segregates. This weakness may sometimes be observed when forging the bottom end of large



Fig. 8—Illustrates What Would be Expected if an Ingot were "Hollow Cast" and Treated Under Normal Casting Conditions.

ingots; the body of the ingot will open out slightly, forming a small annular space with the base of the cone.

Proceeding with a normal ingot mold, in the next example ingot C, (Fig. 6) the metal was cast hot and extreme cooling conditions were effected. The bottom plate or stool was water-cooled while the major portion of the mold was kept hot by gas rings and a gas flame kept impinging on the surface of the molten metal in the mold.

The influence on the development of the bottom cone is of outstanding interest, while the growth of the entire crystalline structure shows an upward sweep, and in the case of steel, the segregates may well be conceived to lie between these marked crystalline boundaries. On comparing this example with the segregate in the



Fig. 9—Ingot Allowed to Cool Normally. Due to Comparative Fast Rate of Solidification Shows Even Distribution of Segrates.



Fig. 10—As Soon as Cast Upper Half of this Ingot was Encased in Incandescent Charcoal for Half Hour While Lower Half was Allowed to Cool Normally.

upper portion of Fig. 1, quite a marked comparison may be noted.

Ingot D, Fig. 7, was cast at normal temperature and a ring, water spray, was applied from the middle-half downward, while the top half was kept hot with a gas ring and the metal kept liquid at the top as in ingot C.

Ingot E, Fig. 8, is included only to illustrate what might be anticipated if an ingot were "hollow cast" and treated under normal casting conditions, and insomuch that it suggests the whole of the segregates would be forced into the center of the wall section.

It might however be conceived, as will be noted later in this paper, that by rapid cooling, and dependent on the thickness of the wall of the hollow cast ingot, segregation can be almost entirely prevented.

Steel Ingots

It will be appreciated, that due to the rapid rate of solidification of steel in very small ingots, as compared with zinc, it is more difficult to retard its progress with a view to promoting segregation. Ingots F and G, Figs. 9 and 10, which are photographs of sulphur prints, do however conform to what might be anticipated, and correspond closely with the results obtained from the zinc ingots. Both ingots were cast from metal remaining in the ladle after casting a 44,000-pound ingot.

Ingot F, (Fig. 9), was allowed to cool normally and due to its comparative fast rate of solidification, shows a very even distribution of segregates.

Ingot G, (Fig. 10), as soon as cast, the upper half was encased in incandescent charcoal for half an hour, while the lower half was allowed to cool normally. In the upper half, a marked tendency is shown in the development of segregation.

Reverting to Heavy Ingots—during the progress of their manufacture and following up the information obtained from the experimental ingots, experiments were made with a view to improving their quality for the purpose for which they were required.

It was thought that by the addition of aluminum to the still liquid discard portion of a large ingot the solidifying point would be depressed and retarded, and thus stimulate the flow of segregates from the main body of the ingot to this area and lead same up to the still more fluid sink-head. Five pounds of aluminum were therefore added to the liquid metal when within six inches of the top of the mold, and casting continued as usual into the sink-head.

It was however found, that by this addition, the freezing point of the serviceable metal well below the portion treated was also depressed, and that segregation was developed rather than lessened. The discard portion of this ingot is shown in Fig. 11. The segregate lines closely follow the orientation in zinc ingot Fig. 7, and the extent of the segregates in the serviceable portion of the ingot can be well imagined.

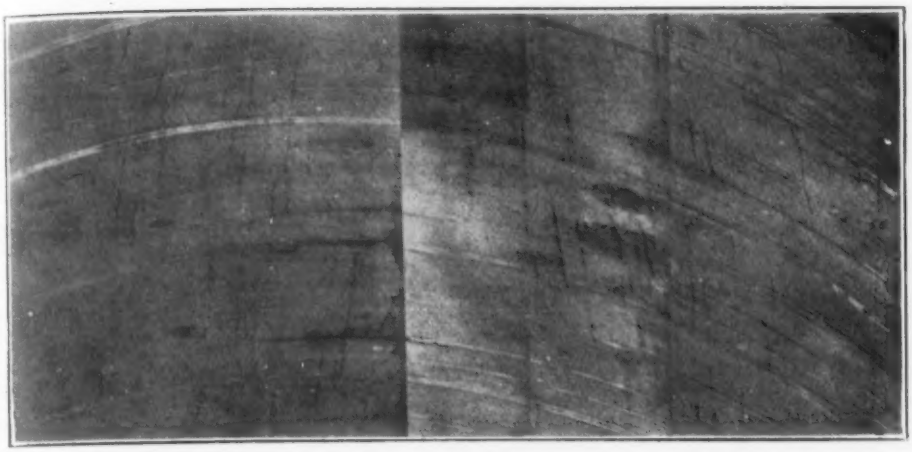


Fig. 11—Discard Portion of Ingot Showing Segregations.



Fig. 12—Ghost Series in Ingot.

Attention might here be directed to the characteristic form taken by the major segregation. Although after its accretion into small masses it would still appear to be free to flow, as shown in Figs. 1 and 2 it is restrained within phase areas. These areas also are mobile and as seen in Fig. 2 although they have been considerably deflected have, apart from an apparent strained action, retained their original relative positions.

On comparing several examples, from ingots of the same class,



Fig. 13—Sulphur Print of Transverse Face of a Large Ingots.



Fig. 14—Split Cores Showing Comparison Between Normal and Accelerated Cooling.

where the casting conditions have been very similar, the upward parallel movement is identical, even to the width of the phases.

The writer suggests the possibility of the temperature in these phase areas being greater than the still fluid ingot center, due to the evolution of heat from each phase during crystalline growth and solidification. This may explain why the segregates are retained between these phases and do not diffuse to the center.

In the manufacture of large hollow forgings, the usual procedure is to remove the top and bottom discard and trepan the core. The latter may be as much as 25 inches in diameter. Provided that on superficial examination, little ghost marking has been detected, it has been considered safe to proceed with the forging. Considerable anxiety has sometimes arisen when machining the bore of such forgings to find well developed ghost marks distributed over a large area. Fig. 12.

The photograph of a sulphur print Fig. 13, taken on the transverse face of a large ingot gives a ready index. The trepanning has followed down through a clean area, between two distinct phases, the primary and secondary segregation. This therefore suggests the policy of running a sulphur print strip across the center and full width of the machined face of such ingots. By splitting and examining the cores further useful information is also obtained.

The two split cores Fig. 14 are for comparison, between normal and accelerated cooling, and show the effect right to the ingot center.

The ingot from which core B was taken was more quickly cooled by spraying compressed air on the mold walls, from numerous orifices in ring-tubes surrounding the mold, immediately after casting and for an extended period.

Another cause of annoyance to manufacturers of large ingots is ingot corner segregation and corner cracking. Examples of each are shown in Figs. 15 and 16. These two defects are first cousins and if not perceptible to the unaided eye can usually be detected by sulphur printing the ingot corners of the discard face. These defects, however, often do not run the full length of the ingot and the worst location generally is about four feet from the base. When the ingots are allowed to go cold, the corners should be ploughed into and examined, as these defects usually are deeply seated and are not disclosed until the corners are removed down to a diameter equivalent to the octagon face of the ingot. If taken direct from the casting pit and cogged, the billet on cooling

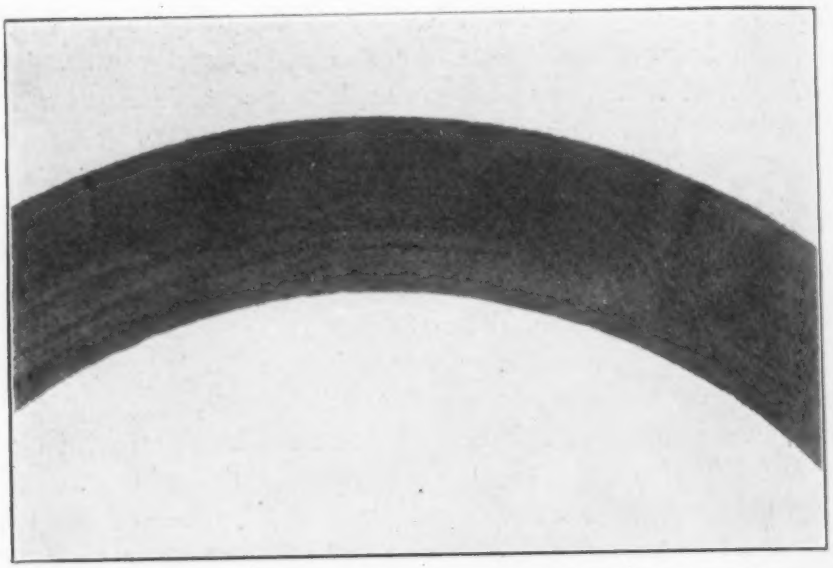


Fig. 15—Portion of Large Ring Showing Cracks. Due to Ingot Corner Segregation.

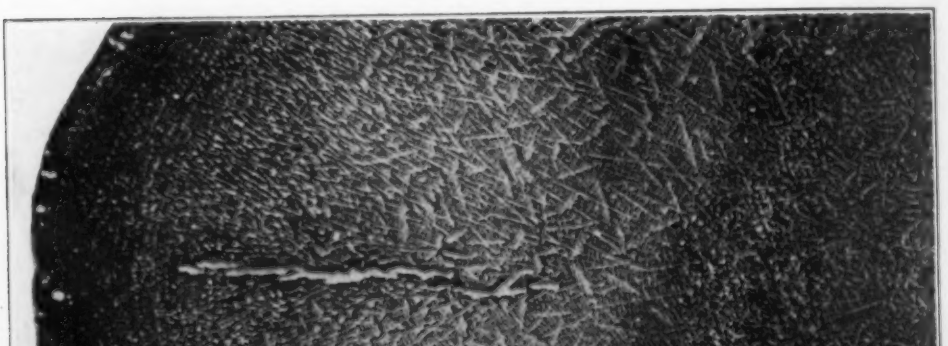


Fig. 16—Corner Crack in Ingot.

should be "banded" about four feet from the top and bottom and sulphur printed. The seriousness of these defects is their deep enclosure, which have been known to carry through to the completed unit and only noticed on the test bed or when serious consequences have arisen due to rupture.

Much has been said and written concerning the advantages and disadvantages of casting ingots with the big end up. This method

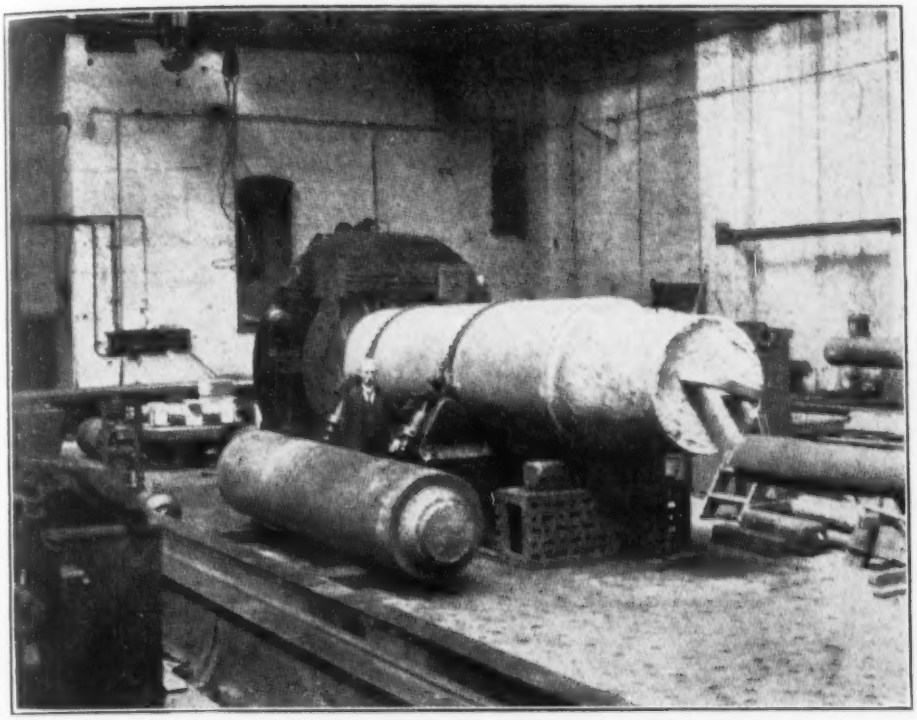


Fig. 17—Large Ingot in Center Weighs 60 Tons.

is unquestionably the best where careful casting conditions are not observed, but equally good and sometimes better ingots are cast, big end down where the casting temperature and rate of teeming are carefully controlled and efficient sink-head provided.

One advantage of this latter method is that the mold can be stripped at an earlier stage, the ingot partially cooled in air, and then through the critical periods, allowed to cool more slowly by dropping over the ingot a refractory lined casing,—anent an old boiler tube. Further, where ingots are to be trepanned for hollow forging, the question of pipe is of little moment. For this same purpose and to remove the risk of refractory materials falling from the sink-head into the mold or steel during casting, it is not uncommon to use unlined sink-heads.

Having outlined a number of the more serious difficulties met with by manufacturers of heavy ingots and particularly those of high quality products, the chief step is to adopt such procedure which will, if not entirely, largely eliminate them.

During the past twelve years papers of very high order have been presented, both in America and England on steel-making proc-

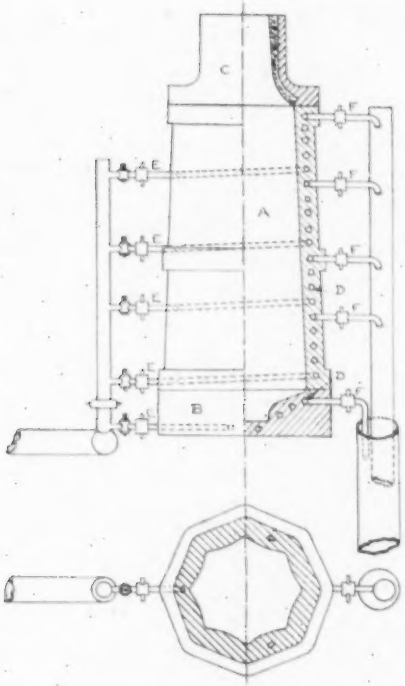
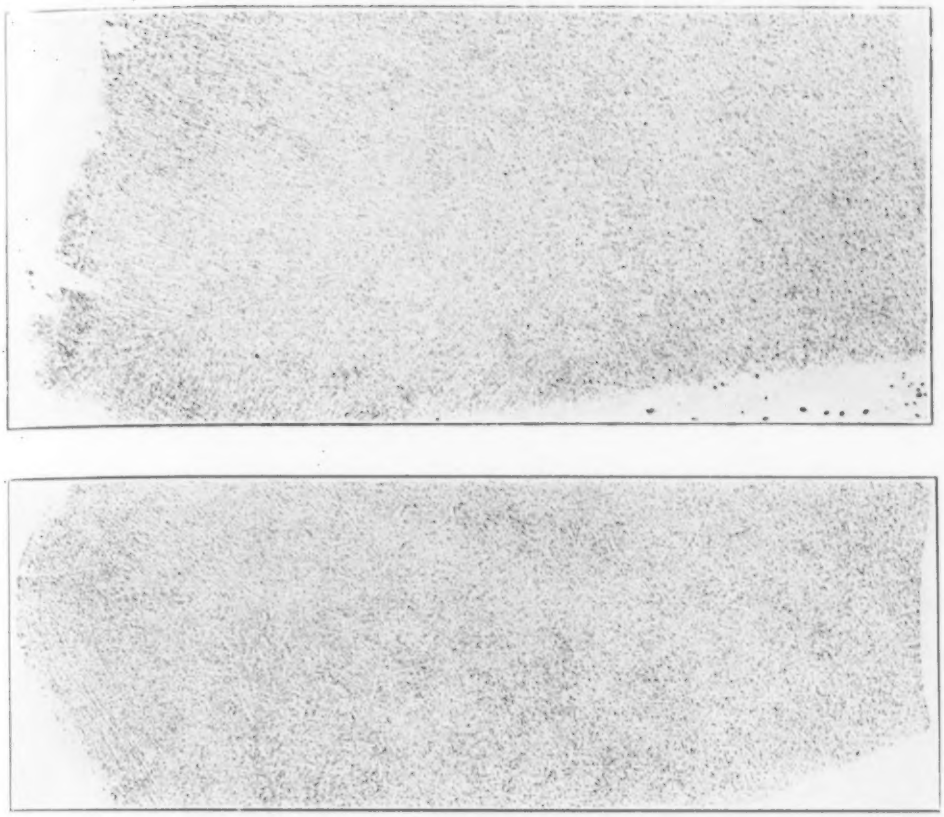


Fig. 18.—Air or Water-Cooled Ingot Mold.

esses, the subsequent manipulation of the liquid metal, and resultant ingots, but there yet remains considerable scope for improvement.

In making high quality ingots, in the first place, the furnace must be seasoned. The first few heats from a new furnace or off a new bottom are never of dependable quality. Modern plants are comparatively easy of control, but apart from the furnace, there are many factors to consider, any one of which may have a marked influence on the final product. Unexpected difficulties may arise, and do arise during the process of melting, with the furnace structure, the bottom, crown or blocks, fluctuation of draught or gas supply, ladle and casting conditions.

Metallurgists and melters, none are infallible, and although all may have the same high quality product as their ultimate objective, may have slightly different methods of attaining that end. Steel plants are run practically continuous, which entail periodic changes of the personnel and in those changes even minor alterations by the oncoming staff may have marked differences on the final product. The writer has known of many heats of steel giving most disappoint-



Figs. 19 and 20—Ink Prints (Considerably Reduced in Printing) Taken From Corner Sections of Disks From Comparison Ingots of Same Cast.

ing results due to a melter, in all good faith, making a minor addition to a slag which took considerable time to elucidate and restore normal results.

Of recent years, experience has shown that by casting these heavy ingots toward the outside of the mold and not in the center, as has been normal practice, marked superior results have been obtained. This method is greatly facilitated by revolving the ingot mold at a comparatively slow speed during casting. In the case of octagon ingots the sonims which always accumulate on the rising liquid surface, are forced to the center and eventually brought into the sink-head. This prevents patches of the sonims sticking to the mold wall and being entrapped in the rising liquid steel, ultimately causing deep surface defects during forging. Further the hot metal in the mold is more equally distributed, giving a less steep gradient from outside to center. This minimizes the risk of corner segregation and corner cracks, reduces the pronounced

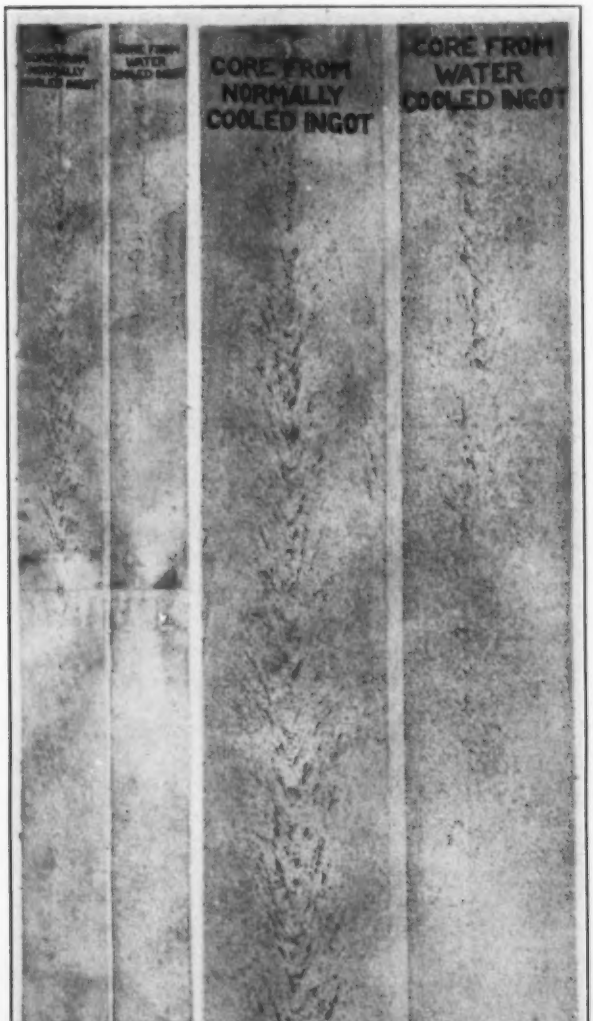
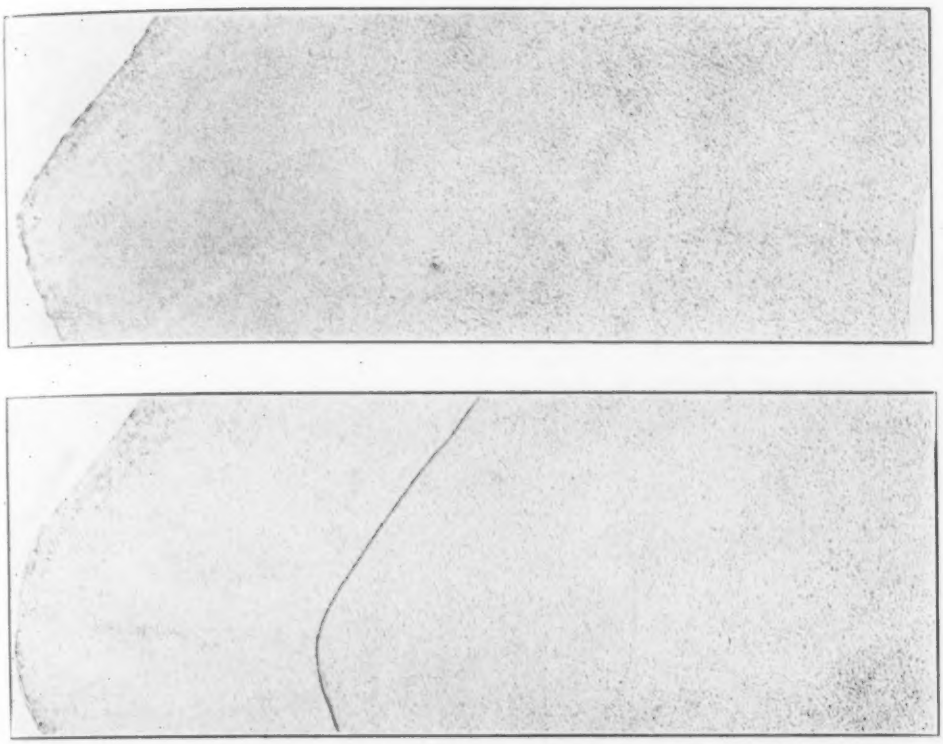


Fig. 21—Photograph of Sulphur Prints Taken from the Cores of Ingots.

dendritic ingot structure and has a marked effect on the distribution of the segregate.

A further development during the past twelve years has been the manufacture of heavy ingots of cylindrical form. (Fig. 17). Many thousands of tons of these ingots have been made without loss, up to 179,000 pounds in weight and larger ones will follow as the existing octagon molds become worn.



Figs. 22 and 23—Ink Prints (Considerably Reduced in Printing) Taken From Similar Disks from Pair of Ingots Cast from Same Ladle, Poured at Normal Casting Temperature.

The advantage of this form of ingot is, that it eliminates all corner defects and the sonims are brought more readily into the center of the mold during casting. It reduces forging costs and in the manufacture of hollow forgings removes the risk of the ends bursting.

Clean melting, rate of casting, and casting temperature of the liquid metal are all important factors and contribute largely to success, but perhaps the most important feature is the rate of solidification. This latter is influenced to a marked extent by the rate of casting and where large masses are involved, efficient means must be devised to accelerate the rate of freezing.

With this object in view, the writer some years ago introduced an air or water-cooled mold, Fig. 18. This constituted a series of steel pipe coils encased in the walls of the mold, which also strengthen the mold, and through which the cooling media is "drawn" and circulated. Some hesitance has been expressed relative to the use of water but if this is "drawn through" on the same principle as applied to the water-cooled tuyeres of the blast furnace there is no

need for reluctance, as in the event of leakage, water would cease to flow and air follow.

It will be noted, the cooling coils are in stages in order that the level of the fluid steel in the mold may be followed up, or the rate of cooling controlled at any desired portion of the mold. The mold may also be retained at any desired temperature until the liquid steel has been cast.

To test out this method, a mold for a 31,000 pound ingot was

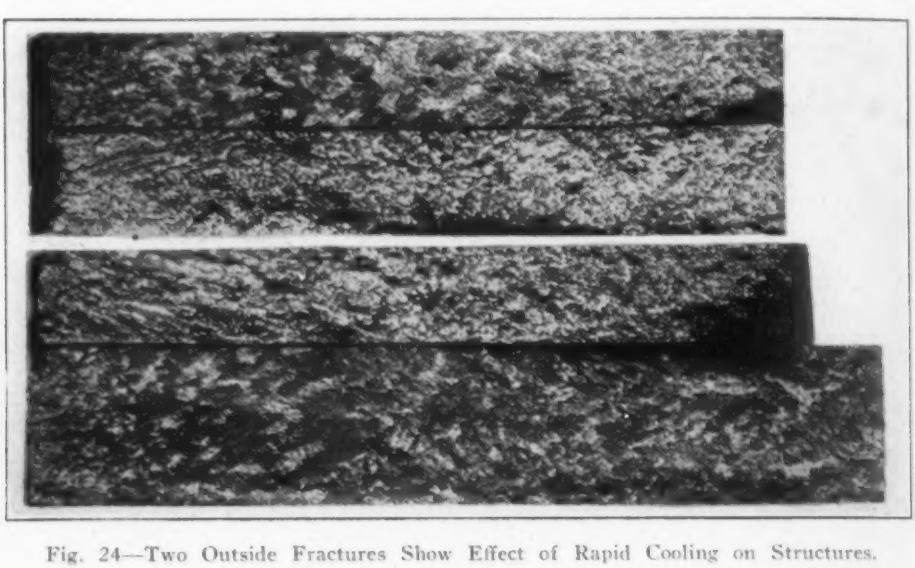


Fig. 24—Two Outside Fractures Show Effect of Rapid Cooling on Structures.

cast with coils encased and from a number of 67,000-pound charges, two 31,000-pound ingots were cast from the same ladle in exactly the same type mold, one being water-cooled while the other was allowed to cool normally. At each cast the ingots were run alternatively, No. 1 in the water-cooled mold, No. 2 in the normal mold, and vice versa.

In every case, during the whole of the casting period and throughout their cooling, it was possible for one to place their hands on any portion of the walls of the water-cooled mold. With the normal mold, in a very short time, the walls became red hot and owing to the radiant heat one could not get within several feet without considerable discomfort.

In view of the type of work for which these ingots were cast it was possible to cut a complete disk from the center of any ingot and also to examine the whole length of any core. Figs. 19

and 20 show ink prints taken from corner sections of disks, from companion ingots of the same cast. "A" cast in the normally cooled mold. "B" cast in the water-cooled mold. These ingots were, intentionally, cast from hot metal to try out the value of the water-cooled mold.

Ingot A, while the upper discard portion was being sawed through, split for its full length,—while ingot B followed through its normal forging operations and produced two highly satisfactory hollow forgings and passed into service.

Fig. 21 is a photograph of the sulphur prints taken from the cores of these respective ingots and the effect of the rapid cooling is demonstrated right to the center of the ingot.

Figs. 22 and 23 show ink prints taken off similar disks, from a pair of ingots cast from the same ladle, but poured at normal casting temperature.

Ingot A from the water-cooled mold, ingot B from the normally cooled mold. Both ingots were forged, and forgings from ingot A passed into service, while forgings from ingot B were rejected due to well developed longitudinal segregate lines, corresponding with the ingot corners,—as illustrated in Fig. 15, being exposed during machining operations.

From the photograph of the fractured disks Fig. 24 and also the ink-prints it will be appreciated the marked effect the rapid cooling has had on the structural condition.

From the analyses taken on the outside of the 16-inch diameter cores, at intervals of 12 inches from the base upward, the following are the average of four, in each case.

CARBON ONLY. ("A" Bottom End)

	A	B	C	D	E	F	G	H	I
Water Cooled	.29	.29	.29	.29	.30	.30	.29	.29	.31
Normally Cooled	.29	.29	.33	.33	.33	.34	.34	.34	.35

With the water-cooled ingot not only was the carbon uniformly distributed but also the elements sulphur, phosphorus and nickel. In the case of the normally-cooled ingot, these elements were migratory with the carbon.

This same stability and migratory movement was shown by the analyses taken from the central disks, from outside to center. Unfortunately these complete analyses, at the moment, cannot be traced.

The demands of the engineer for an ever increasing weight

and a more dependable steel is a progressing factor to be recognized, and the writer suggests it is along the lines indicated above that a solution of these problems, both with regard to segregation and ingot corner defects, will be found.

The rapid development of power generation plant calls for a continual increase in the size of forgings, even if made in alloy steels, and in view of these great masses, and to minimize segregation and other inherent defects some such effective means must be adopted to promote more rapid solidification.

The bursting of rotor bodies while running at enormous speeds, causing death, and destruction of valuable plant is of rare occurrence, as also is the bursting of naval ordnance. Such cases have however occurred and their contributary causes have in a large measure been due to these ingot defects.

The need for equally much care, and along the same lines, is called for in the manufacture of the smaller ingots for service in aeronautics, automobiles and railroad locomotives, on all of which life and limb are dependent.

DISCUSSION

Written Discussion: By W. H. Hatfield, Brown-Firth Research Labs., Sheffield, England.

It was to be regretted that the results of the investigations have had to wait such a long time for publication, for some of their value is minimized by the prior publication of similar data. Most of the results are in accord with the findings of the Heterogeneity Committee of the Iron and Steel Institute, but there are a few items on which I would comment.

In the first place data obtained on small scale ingots, although they are proportioned in all respects to large scale ingots, cannot be relied upon in giving correct information concerning the latter. The mechanism of freezing is dependent on several factors, two of which are (1) the rate of cooling through the freezing range and (2) the temperature gradient across the ingot, and although artificial means may be adopted to make the rates of cooling identical at one portion of the ingot (say the outside), it will not be so at other points. Thus it is not difficult to reduce considerably the amount of segregation in small ingots. It is in large ingots where segregation phenomena are most pronounced and in my opinion, even using water-cooled molds in such cases, the segregation phenomena will not be obviated. There is another danger which must not be overlooked (apart from the risk of contact of the water with the molten steel—a very real danger) and that is with water cooled molds the thermal stress produced by the greater temperature gradient across the ingot are more likely to form hair cracks, particularly in such difficult alloy steels as nickel-chromium or nickel-chromium-molybdenum.

On the eleventh page of the paper it is suggested that there is the possi-

bility that in the areas where segregate occurs, the temperature during freezing may be greater that at the center. This possibility has been discussed by the Heterogeneity Committee and it would appear that this state of affairs is not possible. It must, however, be remembered that the layers adjacent to the freezing wall are rich in segregate and, in consequence, have a lower freezing point than the parent liquor. There is, therefore, the possibility that these layers will actually freeze later than portions of the ingot nearer to the center.

Written Discussion: By John E. Jacobs, U. S. Bureau of Mines, Pittsburgh.

In the second paragraph on the first page of the paper, the following statement is made: "The work was carried through at the expense of one firm, and in view of the high costs involved in surface machining these large ingot sections, this was dispensed with as it was then considered the information required was obtained." From this statement and from the obvious appearance of saw marks on the photographs shown in Figs. 1 and 2, these sections must have been etched without polishing or grinding the surface to be examined.

A short time ago some sections of steel ingots in the "as cast" condition were examined at the Heppenstall Company, Pittsburgh, Pa., in cooperation with the U. S. Bureau of Mines. These sections were cut longitudinally from the top of the hot top down through the exact center of the ingot to a point about 30 inches below the ingot-hot top line. Three sawed sections were etched for 30 minutes in 1:1 HCl at 180 degrees Cent., and photographed. The etch was then machined off and the surface ground before going through the same procedure again.

When the results of these tests were compared, a striking difference was noted in the etch taken on the ground surface and the "as cut" surface. In each instance the ground surface was much more readily attacked by the acid, and it was hard to believe that the two sections were from the same piece of steel.

These observations lead to the conclusion that some standard method for the preparation of surfaces should be established and adhered to whenever etch tests are to be used in a comparative way, and it would seem best to grind the surface before etching.

Written Discussion: By Axel Hultgren, metallurgical engineer, Söderfors Bruk, Söderfors, Sweden.

The author expresses the opinion that the upward sweep of the crystals in the zinc test ingot shown in Fig. 6, as compared with the similar configuration of the segregates in the large steel ingots shown in Figs. 1 and 11, points to an analogous mechanism of crystal growth in both cases. This statement may be questioned on the following grounds.

From the evidence known to him the writer has concluded that:

1. Segregates do not occur in the zone of elongated primary crystals, but in the region of free crystals.

2. Elongated primary crystals are not found to assume the steep slope of the segregates.

3. Elongated crystals, when found associated with segregates—travers-

ing them—are secondary gamma crystals formed by transformation from the delta state and thus their structure cannot illustrate the mechanism of freezing.[1] Any evidence in the author's possession, contrary to the above conclusions, would be welcome.

The zinc ingot sections shown are of great interest also for the reason that they provide beautiful examples of curved crystals grown from the molten state. It has been suggested[2] that curved primary crystals have not been observed in steel ingots—except when deformation has occurred—and that this is due to the pointed shape of the crystal skeletons during their growth, whereas the fronts of the growing crystals of a pure metal are believed to assume a more blunt shape, the crystal boundaries being thus able to accommodate themselves to the changing direction of the isothermals during solidification.

The cause of the segregation shown in Fig. 10 is not clear. Is it not possible that the retarded freezing of the ingot was in some manner responsible for the formation of blowholes, in conjunction with which the segregates formed?

The author refers to the *upward* flow of the segregates as indeed do most authors on this subject. The writer ventures to suggest the possibility that the segregate strings are formed by successive suction *downwards* of the impure mother liquor during the later stages of freezing of the aggregate of free crystals in the intermediate zone.[3]

The author demonstrates the benefit derived in various ways by water-cooling the mold in casting heavy ingots. It may be of interest, therefore, to know that during experiments with 9-inch square ingots of 1.10 per cent carbon steel cast big end up the writer found in one respect the opposite effect of water-cooling: segregates were accentuated hereby.

G. A. Dornin:[4] As so much of this paper refers to large ingots and we have a vice-chairman who has probably had more experience with large ingots than anybody I know of, I think some comments from Mr. Adams would be most interesting. My own experience with reference to large ingots, 20 tons and up, goes back a great many years, and is not very recent.

Round ingots cannot be made rapidly on a tonnage basis, on what we, in this country, consider a commercial basis, that is above a certain size. Round ingots are successfully cast up to about 15, perhaps 18, inches in diameter, by bottom pouring very slowly. The principal trouble that they seem to meet is that the refractories are not good enough. Every now and then we run into an epidemic of surface slag inclusions. If you take a round ingot mold and top pour it very slowly, much more slowly than you can in commercial practice, I think it is entirely possible to cast a round ingot, but my experience in pouring steel in steel plants is that you have about 30 minutes,

[1]Hultgren, "Crystallization and Segregation Phenomena in 1.10 per cent Carbon Steel Ingots of Smaller Sizes," The *Journal* of the Iron and Steel Institute, No. II, 1929, p. 81-85.

[2]Hultgren, loc. cit., p. 84.

[3]Compare Hultgren, loc. cit., p. 102-103, where a similar explanation is given for so-called structure in 12" ingots of 1.10 per cent carbon steel.

[4]Consulting and metallurgical engineer, Gathmann Engineering Co., Baltimore.

perhaps a little over, in which to pour a heat of steel, irrespective of size. Make the time 45 minutes. If you are pouring a 100-ton heat in 3-ton ingots, you have roughly 30 ingots to the heat, and you have to pour each ingot in 1½ minutes, unless you pour a number at one time, and in that time I do not think the skin that forms is heavy enough to withstand the internal ferrostatic pressure. As Mr. Ashdown says, these troubles you get from casting round ingots are surface troubles. They come to the surface and you can get them out, but I can never get any one to do a thing which they know before-hand is going to make them go into chipping cost. Chipping cost in this country today, in a well managed tonnage plant (I am not talking about the special plant that makes largely alloys that run to tremendous chipping costs) will eat up an amount equal to 10 per cent of the earnings of that plant. To this cost add surface rejections, which also take about 10 per cent of the earnings. So when I say that you cannot cast round ingots I do not mean that it is not possible to cast round ingots, but I mean that the casting of round ingots at practical rates of pouring is, in my experience, not possible.

P. A. E. Armstrong:[5] Round ingots is a matter that I have been working on for some time. Some trouble has been experienced in practice due to cracks under the head, due to the ingot hanging up in the mold. This difficulty is, of course, the result of the design of the sink head, and has nothing to do with the shape of the mold. The objection, raised by one of the speakers, that round ingots were not successful because of insufficiency of skin to hold up the molten metal of the center, may be an objection but can hardly be laid entirely to the shape of the mold.

The thickness of the chilled or solid skin in a given period is purely a function of how quickly the heat is taken from the surface of the molten metal. If the molten steel is poured into a sand mold the chilling effect is obviously slower than would be the case if the metal was poured into a chilled mold such as the regular cast iron ingot mold.

The round ingot mold for producing round ingots is best made of copper. Simply because cast iron has been used for a great many years is no reason for its continued use. The thermal conductivity of cast iron is quite low compared with that of copper. Therefore, surface cutting of the ingot mold made of cast iron is an easy matter, due to the low melting point of cast iron, and the fact that the thermal conductivity of the cast iron is so low that a high temperature can be imparted to the surface of the cast iron causing sufficient local heat to produce fusion; whereas the thermal conductivity of copper is such that the molten steel will not locally melt the copper surface of the ingot because the heat can be taken away from a highly localized area at a speed which will prevent fusion of the surface copper. Naturally this is accompanied with the necessity for bulk or auxiliary cooling.

There is, obviously, no chance of heating the copper mold in its entirety any hotter than the division of the total heat of the molten steel by the total weight of the ingot cast and its mold. 1000 pounds of ingot metal at 3000 degrees Fahr. poured into a copper mold weighing 1000 pounds could not result in a greater total heat for the mold and ingot of more than 1500 degrees

[5]Consulting metallurgical engineer, 875 Park Ave., New York City.

Fahr. Obviously this figure could not be reached due to the surface cooling present.

The copper mold need not be given a wash, although I have always done so except for experimental runs. I find that aluminum oxide mixed with sufficient clay to make it stick is a very admirable wash, and it sticks nicely to the copper, particularly if the mold is not too hot when it is applied. Heating to dry out the wash is, of course, an obvious necessity.

If the copper mold is water-cooled, a water wash is a little more difficult as means must be taken to dry it. The oxide that is present on the surface of the copper is no detriment although it is, of course, not much more than color and has no particular thickness, but it is a sop to conscience if some sort of coating is required on the surface of the ingot mold. Therefore, swabbing of the copper mold is a matter of taste.

It is obvious to all that a mold cast large end upwards (if it does not hang at the top, which it should not do in any case) is going to stand up on the foot. Therefore, directly the metal solidifies it shrinks away from the wall of the mold, and the thermal conductivity of copper, or anything else, it not going to have a great deal of effect due to the air gap between the solidifying ingot and the mold wall.

If this type of mold is to be employed the copper mold will still be a paying proposition because of the practical inability to cut it and its tremendous durability. If it is water-cooled, which is not dangerous, copper lends itself to the design of a true repetition mold. Water-cooled cast iron may or may not have a tendency to fracture when hot metal is poured against it, but I would feel happier with the copper where the chance of fracture is well nigh impossible.

However, if copper is to be employed, the design of the mold might just as well be made properly and its great thermal conductivity made use of. This calls for another departure from "time honored" practice, which, I venture to say, is about all you can say about some practice. The ingot mold, and I most distinctly favor the round type, should be made of copper, water-cooled, and the foot should be mechanically arranged so that immediately after initial freezing has progressed far enough to give a strong crust the foot can be dropped sufficiently far so that the solidifying ingot is kept in constant contact with the mold wall. Chilling can then progress at a fast uniform rate which will not cause undue piping, prevent segregation to a large degree, support the side wall crust, and cut out surface oxidation of the ingot. Ingots cast in such a mold, particularly if swabbed with the aluminum oxide before referred to, will come out with a bright shiny surface if allowed to cool down to black heat or lower. The ingot mold of this type can, obviously, be swung on trunnions and the red hot ingot dropped from its mold by inverting the ingot mold. There is some mechanical wear of the mold due to the sliding ingot, but the wear is very little.

In designing a mold of this character, care should be taken not to use a foot made entirely of copper, although, at first thought, it would seem obvious that if the ingot mold is to be made of copper so should the foot. The sliding foot or plug at the bottom of the ingot mold, if made of copper,

will freeze about the second time it is used due to the well known characteristic of copper to hang upon itself directly if one piece is slid over another dry. The plug should be made of steel, which, due to the cooling effect of the water-cooled copper mold, will freeze the molten metal at such a quick rate that no trouble will be experienced with stickers. However, with large size ingot molds it is desirable to fit a heavy copper insert in the steel plug or shroud the copper plug with a steel tube.

I feel well satisfied that in the manufacture of alloy steels, where surface imperfections and interior difficulties are the order of the day, copper molds of the type I have described, (which I think were originated by me) will be the molds of the future. Their adoption will depend upon how fast "time honored" practice will give way to efficiency.

I am, of course, aware that I am not old enough to have been the first person to have poured molten steel into a copper mold but my remarks are not directed to just that.

EMIL GATHMANN:[6] I just wish to state that for 15-ton ingots we employ 15 per cent sink head, and the actual crop (the sink heads are being cropped 12 per cent) gives an absolutely sound ingot on the structure. We have never tried these big fellows that you are talking about, so I do not know much that I can say. But with the big end down we find that unless you have a very short ingot relative to its diameter it does require very excessive sink head.

J. M. ADAMS:[7] That portion of Mr. Ashdown's paper on "Steel Ingots", suggesting the possibility of casting steel ingots in water-cooled molds, is quite interesting and might be applied to some advantage where small sized ingots are concerned. It is doubtful, however, whether it would be possible to apply the principle to large ingots such as those of 100 to 200 tons weight. On the other hand, much can be accomplished without recourse to such a drastic expedient as a water-cooled mold, by altering, within perfectly practical limits, the proportions of the chill, taper, and design of the sink head, as the ingots increase in weight.

In this connection, i. e., ingot mold design, there is one point which might be given more attention, namely, that an ingot mold giving a perfectly sound and minimum segregated ingot of steel of one analysis will not necessarily give a proper ingot of steel of another analysis. Failure to appreciate this point has caused considerable trouble in a number of instances.

It is to be regretted that so little care has been taken in preparing the surfaces of the samples shown in the paper representing deep etching and sulphur printing. No reliance can be placed on the results obtained. Experience has shown that the greater the surface, the more care is required in the preparation of surfaces for deep etching and sulphur printing.

Author's Closure

In reply to Dr. Hatfield, Chairman of the Heterogeneity Committee of the British Iron and Steel Institute, the author much appreciates his contribution to the discussion. He agrees that, due to his inability to obtain permission

[6]President and chief engineer, Gathmann Engineering Co., Baltimore.

[7]Superintendent, special products department, The Midvale Co., Nicetown, Philadelphia.

to publish at an earlier date this data, some of its value may have become minimized by prior publication of the British Committee. As, however, much of this work was done fifteen years ago it is interesting to note it is in accord with the recent findings of that Committee. From the results of those early investigations much progress in private capacities with large ingots has been made and the paper introduces several features hitherto not published which indicate definite lines for marked improvements.

It is agreed that small scale ingots do not give a true interpretation of the freezing of the large ones, but by employing the metal from the same ladle and following as far as was practically possible the conditions of freezing of the large ingots they are more strictly comparative than are experiments with wax, and they *do* indicate the same conditions as are found in the heavy types.

The author has shown by comparison with large ingots cast from the same ladle that by accelerating the rate of freezing segregate phenomena is to a great extent prevented. This is proven in the analyses by almost total absence of both negative and positive segregation and also, by sulphur printing, the absence of sulphide segregation.

The assumed danger in the use of water as a cooling media under the conditions submitted is very small, provided reasonable precautions are taken. The mold before use, on each occasion, should be subjected to water pressure test, further the gravity flow of the water together with the light pressure generated by the hot metal would restrict the flow of water or steam within the coils. The alternative is the employment of compressed or suction air through the coils. As proven in the paper any method which will accelerate the rate of freezing, minimize segregation, and reduce dendritic structure is of material advantage.

Replying to the point raised on the temperature gradient across the ingot giving rise to thermal stresses, it would appear Dr. Hatfield has assumed the ingot mold as being filled before the cooling effect is brought into operation. Were that so it is agreed the temperature gradient would be steep. A glance at the mold shows the cooling coils arranged in four stages and as stated in the paper the cooling effect is progressive with the level of the molten metal from the bottom upward.

In the freezing of large ingots too much direct comparison is often made with conditions controlling the smaller ones, whereas in the early stages the casting and freezing conditions affecting large ingots are totally different and the following essential points overlooked.

In the first place due to the large cooling surface area of the ladle bottom and a subsequent rest of the molten steel in the ladle, the steel in the bottom is of considerably lower temperature. This on casting, falling on to a comparativly cold mass of metal assisted by the cold walls of the mold solidifies at a comparatively quick rate which with the water-cooled mold is accentuated by the use of the lower cooling coil. The oncoming metal is quickly lowered in temperature over the whole surface and this condition is progressive throughout the casting. The temperature gradient across the ingot, under these conditions is therefore considerably less than in a normally cast ingot. If it is gen-

erally accepted that where the temperature gradient becomes small equi-axial crystals are formed, the temperature gradient across the water-cooled ingot must be small as the dendritic structure is absent and replaced by equi-axial crystals.

All ingots cast in water-cooled molds were of nickel-chromium steel, every ingot cast was forged and every forging made was used for naval ordnance in the form of, either torpedo air vessels or guns and every forging was accepted and went into service. It is well known that forgings of this character receive the most critical examination and severe testing. The suggestion of hair cracks may therefore be discounted.

The author would have appreciated Dr. Hatfield's opinion on the prevention of the formation of dendrites by accelerated cooling as, so far as he is aware, all theories so far advanced suggest, accelerated cooling promotes their development.

Concerning round ingots the author's statements are not in accord with the views of the Heterogeneity Committee and here again Dr. Hatfield's comments would have been appreciated.

Regarding the question of phase segregation, Dr. Hatfield states, there is the possibility that these layers will actually freeze later than portions of the ingot nearer the center. The author suggests that they freeze at a much earlier period than any portion of the center and would refer him to Dr. Arnolds' paper before the Mechanical Engineers, London, 1915 in which he gives a striking example of the early formation and freezing of the mass segregates.

An ingot of 127,680 pounds in weight (57 tons) burst out at the bottom five minutes after casting and bled 38,000 pounds (17 tons) leaving an upper hollow casing, and attached to the protruding crystals in each octagonal corner were a number of ghosts $\frac{5}{8}$ inch diam. by 9 inches long. That I think is sufficient evidence. Arnold states that it had been an accepted faith that ghosts froze last, but this proves conclusively that they freeze first at many degrees above the main freezing point.

The instance cited by Mr. John E. Jacobs is the first the author has known of anyone attempting to obtain a true interpretation of the structural condition by an acid attack on a roughly sawn face. It is appreciated generally that an acid solution applied to a roughened surface will develop local attack and pitting. For an etch examination a perfectly smooth surface must always be prepared which already is a recognized practice.

On an examination of the illustrations in the author's paper it will readily be detected which are the roughly sawn faces and these only, are photographs of sulphur prints. Although even for this it is not commendable the major segregation is readily observed and in these instances that alone was sought.

For the illustrations Figs. 19, 20, 22 and 23 the surface of these specimens was highly polished before etching and the detail in these illustrations has been lost due, as stated, to the considerable reduction in size from the originals for reproduction. Care has been taken for reproduction in the TRANSACTIONS to obtain greater detail in these reduced prints.

In view of Mr. Hultgren's recent published work on the solidification

of ingots, although of smaller dimensions, his views are greatly appreciated. With regard to Zinc Ingot No. 6. I think here he has misread me. Although I allude to the segregates following a similar configuration I do not infer that the steel crystallizes in that same directional course. All the evidence in the paper is to the contrary, in that it freezes at right angles to the mold walls. A striking example of this is given by the succession of contour lines in Fig. 16. These lines correspond with the branch crystals thrown out from the main stems irrespective of the directional relation of the main stems with the mold walls. If it were possible to retain the steel in a fluid condition at high temperature, as one can the nonferrous metals, there is little doubt its directional freezing could be similarly affected. By the consistent configuration of the segregates in all large ingots similarly cast, as in Fig. 11 the evidence is very strong that the freezing takes place in phase stages. It is agreed that apart from, in the areas of planes of weakness, segregation does not occur in the zone of elongated primary crystals. With regard to points 2 and 3 I am disposed to agree. The ingots Figs. 9 and 10 having been cast from the last metal in the ladle of a 44,000 pound charge, well known to be enriched in segregates, by promoting the rate of solidification it shows that these segregates can be arrested in a fine state of divisicn where they are less harmful as compared with the concentrated segregates resulting from restricted cooling.

Regarding the directional movement of the segregates, in the author's opinion the primary segregates tend to move upward as indicated in Figs. 1 and 2 but that movement is very limited as they unquestionably freeze while the major portion of the steel is still quite fluid, as stated, in reply to Dr. Hatfield.

The orientation of the segregates in the central axial portion of the ingot certainly suggests a downward movement due presumably to the final contraction and central pressure from the metal descending from the sink-head. Much of this axial segregation is often due to the metal in the sink-head being enriched in segregates.

In replying to Mr. Dornin, I think he has modified his views very much with regard to round ingots because the statement in the paper is made in quite general terms and it does not apply to ingots of any particular size. Mr. Armstrong has just emphasized a big advantage of the round ingot of the small type. The great advantage of the round ingot cast in the alloy steel is its readiness for machining prior to rolling or forging or for any drop stamping and upsetting work. One of the many features of the two papers I think has been the reversibility of the mold. Perhaps I can give a little enlightenment on how to make round inverted ingots. (Illustrates on board). We will take it as representing either 50 or 100-ton ingots and the ingots may be cast from two heats. The mold stands on a heavy base. If you can use an extra base, so much the better. (He draws first ladle and second ladle above, on board). The ingots are cast from two heats, two furnaces, and the temperatures are slightly modified. The first furnace tapped is the one of the slightly lower temperature. The ladle is brought out and put onto a carriage over the mold. After about

half the charge has run down from the first ladle the second one is opened into the first so that the ferrostatic pressure is fairly constant. The nozzle in the first ladle is magnesite so that there is no wash. That maintains practically a constant size the whole way through. The nozzle in the second ladle usually is of clay. Assuming the first one to be an inch the clay is an inch and a quarter so that the clay is opening out slightly, and the level of the metal is kept practically constant. It may rise just a little. The charge from the first ladle is all deposited into the lower portion of the ingot, and with the steady flow one gets the hotter metal gradually rising in the top half and filling the mold. Further, the hot metal is chilling quickly here, so there is practically no segregation. That is the general principle of the method. I can assure Mr. Dornin and anyone else that one can obtain a perfectly solid ingot and the proof is that rotor bodies have been made from ingots of that form and the cores examined and passed A 1.

I would like to raise another point in connection with Mr. Dornin's statement. I would like him to advise me of any ingots that have been sulphur printed, big end up, or any other way, that do not show the axial segregation, if that is what he was referring to as indicating unsoundness. I have seen many large ingots sulphur printed and never remember seeing on that has not showed the central sulphide segregation. As I pointed out, that may be very largely due to the segregate descending from the head and not actual segregation from the ingot itself.

G. A. DORNIN: Are you using a sink-head?

H. H. ASHDOWN: Yes, the sink-head would be about 22 to 25 per cent of the weight of the ingot.

G. A. DORNIN: That would not be commercial to use that big a sink-head.

H. H. ASHDOWN: You cannot guarantee an ingot of that size without approximately 20 to 25 per cent in the sink-head. Of course you have to generally speaking. For special work it is specified that you shall remove a certain amount from the head of the ingot, say thirty per cent.

You will find on all the big ingots, whether it is big or small end up, the percentage of sink-heads will run around 20 to 25 per cent. I would refer you to the reports of the British Iron and Steel Institute. The small ingots generally show 15 per cent.

Mr. Armstrong's contribution on water-cooled round ingots is of much interest mainly from a point of view of the substitution of copper in place of cast iron for the material of the mold. The author agrees with the points of advantage claimed over cast iron in-as-much that it has greater conduction properties, is less liable to fracture and produces cleaner skin ingots, but it is a question whether its initial cost is counterbalanced by the saving effected which only can be proven over an extended period.

With small ingots the falling stream of liquid steel can usually be well directed on to a hard center stub but with large ingots and the longer fall of the stream it is less certain of striking the base center and any wash action on the lower walls or base would speedily reduce the life of the

copper mold. This to some extent might be met with a copper body and cast iron stool.

The primary object of the small round ingots for alloy steels is its form for rapidly machining surface defects before forging or rolling. If the copper molds produce ingots with perfect skins other forms of ingots might with advantage be made.

The water cooling of these small ingots will be of much interest to many steel manufacturers of special alloy and tool steels who have been experiencing difficulties due both to segregation and dendritic structure and Mr. Armstrong's views on the rapid chilling effect on the structural condition would be of much interest.

Many thousands of 2000 to 3000-pound alloy steel round ingots have on a tonnage basis been produced from cast iron molds without any difficulty and shown little segregation. In all cases the surfaces have been pinholed and required skinning. All these materials have conformed to drastic specifications for service in airplanes, automobiles and other exacting conditions.

Mr. Gathmann's query relative to the percentage of sink-head allowed for large ingots as compared with small may be explained that for special purposes it often is specified that ingots shall have thirty per cent discard removed from the top end. This leaves the steel manufacturer free to provide a sink-head with say twenty-two to twenty-five per cent and the remainder for removal from the ingot body.

With a well designed mold effecting a progressing rate of freezing upward, a careful control of the casting temperature and rate of pouring, particularly restricting this toward the top end of the ingot and the sink-head there is no difficulty in producing perfectly sound ingots of large type with the big end down.

Mr. J. R. Adams' contribution on the subject of water-cooled molds for large ingots is much appreciated. In the preprint paper due to error the experimental ingot is shown as 21,000 lbs. but should have read 31,000 lbs. A 15 ton ingot is a reasonable weight to start experimenting with and can hardly be classed as a small ingot and as this has proved entirely satisfactory it is for those interested to develop the conditions to meet the greater sizes.

However much one may alter the proportions of a chill mold, as cast iron relatively, is a poor conductor of heat it is impossible to prevent selective freezing in large ingots and therefore there must be considerable segregation. The design of the sink-head can only assist in feeding the ingot with a view to shortening the pipe and as pointed out at the meeting the greater the mass in the sink-head the slower the cooling and greater the segregation and this enriched segregated material descends into the body of the ingot often forming a portion of the final product.

If, as already pointed out, the use of water is considered too drastic, the alternative is the use of either compressed or suction air.

The question of mold design for different types of steel has I think already received much attention as shown by the reports of the ingot committee of the British Iron and Steel Institute.

DEOXIDATION AND MOLD CONDITIONS ON THE TENSILE PROPERTIES OF CARBON STEEL CASTINGS

By J. V. McCrae and R. L. Dowdell

Abstract

Foundrymen occasionally experience difficulties with steel castings in meeting ductility specifications of 24 per cent elongation and 35 per cent reduction of area if the steel is deoxidized with aluminum shortly before pouring the castings. Sound castings can usually be obtained in dry sand molds without the addition of "deoxidizers" immediately before pouring.

Green sand molding is generally desired but porosity, as "pin-holes," often occurs. Aluminum and other "deoxidizers" studied eliminated porosity but lowered the ductility. The mold conditions are probably the important variables responsible for pin-holes. A sufficient amount of "deoxidizer," however, should be in the steel at the time of pouring to deoxidize the steel from the gases evolved from the moisture contained in the mold.

I. Introduction

THE problem of obtaining sound steel castings in green sand molds with the most desirable mechanical properties has long been studied by steel foundrymen. Frequently steel foundrymen are called on to meet specifications[1] in which the minimum tensile strength is 60,000 pounds per square inch; the minimum yield point 45 per cent of the tensile strength; the minimum elongation on a 2-inch gage length 24 per cent; and the minimum reduction of area 35 per cent, these properties being determined on a 0.505-inch diameter tensile specimen.

These specifications can be generally fulfilled by the foundry-

[1] U. S. Navy Department Specifications 49S1G, April 1, 1925, Steel: Castings: (Class B).

Publication approved by the Director of the Bureau of Standards of the U. S. Department of Commerce.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. Of the authors, J. V. McCrae (now deceased) was research associate at the Bureau of Standards for the Steel Castings Development Bureau and R. L. Dowdell, a member of the society, was metallurgist at the Bureau of Standards. He is now Professor of Metallurgy, University of Minnesota. Manuscript received April 22, 1930.

men if dry sand or surface-dried molds are used, but green sand molding is desired in many cases. In using green sand molds, it is frequently found that small blow holes, usually known as "pin holes," are encountered at fillets and similar positions on the surfaces of the castings. There may be gases in the steel itself which cause some of this trouble, but most of the trouble seems to be due to the moisture in the green sand molds. These pin holes are apparently caused either by the moisture itself, as steam, or its dissociation products or both.

The usual method for pin-hole elimination is to add aluminum to the steel. This is very effective when the aluminum is added in amounts of about 1 pound to one ton of molten steel. While the addition of aluminum will usually eliminate the pin hole trouble, it will generally reduce the ductility of the resulting castings as indicated by both the percentage elongation and the percentage reduction of area.

Steel castings can usually be made to pass the above specifications by any means which reduce the moisture content of the molding sand which comes in contact with the castings. In the past most foundrymen have used green sand molding conditions and have eliminated pin hole trouble by adding aluminum. With average foundry conditions, it is easy to make sound castings in green sand molds by the use of aluminum or some other good deoxidizers although, as just stated, the ductility of the resulting castings is impaired. This effect persists even after heat treatment consisting of either normalizing or annealing from 1650 to 1700 degrees Fahr. (900 to 925 degrees Cent.) followed by reheating to temperatures just below the Ac_1 critical point or about 1300 degrees Fahr. (705 degrees Cent.).

The production of high grade steel castings free from porosity, with high mechanical properties and at as low a cost as possible, is a problem of very considerable industrial importance. When this problem was brought to the attention of the Bureau of Standards, it was considered a suitable subject for study by this Bureau. The United States Government is a large purchaser of this type of material, and it was felt that this would be both a suitable and desirable subject for investigation.

The work was undertaken, on the research associate plan,[2] in co-operation with two founder members of the association which is now known as the Steel Castings Development Bureau.

[2]Bureau of Standards Circular No. 296.

II. Previous Investigations

A great deal of a rather general nature has been written on steel castings. Most writers agree that if steel is deoxidized in the ladle a sufficient time should be allowed before pouring in order to give the deoxidation products and general nonmetallic inclusions a chance to escape from the metal by rising to the slag. Some writers are of the opinion that sound castings can readily be obtained in green sand molds without the use of aluminum or special "killing" agents. Most authors of steel casting articles state, however, that aluminum is generally used in as small a quantity as is necessary.

It has been shown by Herty[3] and his co-workers that iron oxide dissolves in iron as FeO, the solubility of iron oxide in iron being 0.304 per cent oxygen at 2910 degrees Fahr. (1600 degrees Cent.) and 0.452 per cent at 3090 degrees Fahr. (1700 degrees Cent.). The distribution of iron oxide between slag and metal was determined under lime-iron oxide slags and was reported also to be the same under pure iron oxide slags. The experimental work was done in connection with basic open-hearth furnace practice. Later work by Herty and Fitterer[4] has shown that the melting point of FeO is between 2470 to 2510 degrees Fahr. (1355 to 1377 degrees Cent.). They have also concluded that coalescence of nonmetallic particles in molten steel does not take place unless the particles are in the fluid state. The fluid particles must be brought into contact for coalescence to occur.

An interesting paper has been published by Young.[5] In the study of steel made in a side blown converter, he found that the use of titanium in any form decreased the elongation and ductility and was to be avoided in quantities greater than 1 or 2 pounds of titanium alloy per ton, depending on the titanium content of the ferro carbon-titanium alloy used. The untreated steel was reported to be superior in tensile properties to the treated steel and that titanium had been used as a precautionary measure. He also reported that the ductility

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[3]C. H. Herty, Jr., J. M. Gaines, Jr., B. M. Larsen, "The Physical Chemistry of Steel Making: The Solubility of Iron Oxide in Iron," Bulletin 34, 1927, Carnegie Institute of Technology, Pittsburgh.

[4]C. H. Herty, Jr., G. R. Fitterer, "The Physical Chemistry of Steel-Making: Deoxidation with Silicon and the Formation of Ferrous-Silicate Inclusions in Steel." Bulletin 36, 1928. Carnegie Institute of Technology, Pittsburgh.

[5]E. R. Young, "Making Steel Castings to Specifications." *Transactions*, American Foundrymen's Association, Vol. 29, 1921, p. 245.

of the steel castings was decreased by the addition of aluminum and of an alloy of aluminum and titanium.

An interesting paper[6] has been published in which the author states: "He has always used aluminum in small amounts as a safeguard, principally in green sand castings. He is satisfied that if added in small amounts to previously well made steel, aluminum tends to confer on the steel a certain degree of capability to resist the unbalancing effects of mold gases and water vapor." In the discussion of this paper it seemed to be the general opinion that aluminum "deoxidizers" were necessary in the production of small castings in green sand molds, but with large castings in dry-sand molds, aluminum was not necessary if the steel was properly made in the furnace.

III. Plan of Investigation

1. Preliminary tests

The first series of tests was planned with the aim of determining the extent of the reported difficulties. In the early plans, it was decided to study consecutive heats from the different foundries. For this preliminary work, 100 consecutive heats were produced by Company "A," 32 heats (not all consecutive) by Company "B."

The conditions of manufacture were recorded in detail in only the first foundry. These results are given later in detail. The lack of detailed information on the heats from the other company was not due to lack of desire to co-operate but rather to the difficulty of securing adequate information under conditions of normal production.

The seriousness of the question of low ductility was well brought out by these preliminary experiments and on discussing this problem of low ductility in steel castings with steel mill operators, it was found that this same problem is quite common with rolled and forged steels as well as with steel castings. It was evident, therefore, that in this study of steel castings all possible phases of the subject that might influence the results should be included. With this in mind the plan of study was outlined to include the effect of varying ordinary foundry conditions, such as green and dry sand molds, miscellaneous variables such as segregation, design of test castings, heat treatment, cast-

[6] F. A. Melmoth, "Variables in Steel Foundry Practice." Exchange paper from Institute of British Foundrymen. *Transactions*, American Foundrymen's Association, Vol. 36, 1928, p. 323.

Table I

General Foundry Practices at the Two Plants in the Production of the Test Castings.

(All castings were poured over the lip of the ladle).

	Acid Electric Steel Foundries		
Plant	B	A	A
No. of heats	30	100	29
Type of furnace	Heroult	Graeves-Etchell	Graeves-Etchell
Furnace capacity (as used)	2½ ton	2 ton	2 ton
Initial Charge:—			
Shop scrap, per cent	40	40	40
New scrap, per cent	60	60	60
Time:—			
Melt	1 hr. 10 min.	1 hr.	1 hr.
Heat	2 hrs.	1 hr. 15 min.	1 hr. 15 min.
Carbon on melting (average per cent)	0.28	0.20-0.30	0.20-0.30
Additions:—			
Iron ore	60-70 lbs, with initial charge	As needed after melting	As needed after melting
Lime	Usually, after melting	6 lbs. before Si addition	6 lbs. before Si addition
Carbon	As needed, (as coke or pig)	As needed (as pig)	As needed (as pig)
Ferromanganese (80% Mn) when added	55 lbs. 3-5 min. before tap	30 lbs. in 2-ton ladle	35 lbs. in furnace just before tap
Ferrosilicon (50% Si) when added	As needed, 10-20 min. before tap	As needed, 10-15 min. before tap	As needed, 10-15 min. before tap
Aluminum, per ton of steel	1.2 lbs.	1 lb.	1 lb.
Where made	large ladle[1]	shank ladle[2]	large ladle[1]
Test Pattern	2-leg[3]	1-leg[4]	1-leg[4]
Heat Treatment	1600-1650°F., 6 hrs.; furnace cool to black heat	1650°F., 2 hrs. soak; air cool	1650°F., 2 hrs. soak; air cool

[1]Ladle contains the entire charge.
[2]Ladle contains only a part of the charge.
[3]Fig. 2.
[4]Fig. 1.

ing temperature, character of scrap, various deoxidizers and deoxidation methods and various mold conditions.

2. *Foundry practice used in the production of test castings*

a. Melting. The typical melting practices used by the two foundries are shown in Table I. These practices which did not differ materially were closely adhered to with only slight changes which will be stated as the results of this investigation are discussed. A study of Table I shows that the two foundries used what is considered to be the normal melting practice on steel castings. All test castings used in this investigation were made in the two co-operating foundries.

The commercial production of acid electric steel castings is practically a plain melting operation with no attempt to refine to the de-

Table II

Characteristics of Green Sand Facings Used by Foundries in the Preparation of the Test Castings

Foundry	Moisture Per Cent	Permeability cm³ Air Per Minute per cm² Sand	Compressive Strength Lbs. Per Sq. In.	Sieve Analysis Per Cent of Sand Retained on Mesh[1] 6	12	20	40	70	100	150	200	270	Pan
A	4.69	173.0	5.4	.15	.05	1.30	35.60	52.95	6.80	1.00	.50	none	.50
B	3.13	172.0	2.7	none	.25	3.70	46.25	38.20	9.20	1.75	.30	.08	.25

[1]U. S. Standard.

gree which is practiced with basic lined furnaces. In the production of steel castings, the scrap is selected so that its phosphorus and sulphur content are usually below 0.05 per cent each. The charge is melted; brought to the desired composition by suitable additions; deoxidized, either in the furnace or the ladle or both; tapped and poured into castings.

b. Molds. Both green sand and dry sand molding were used by the plants in the preparation of test castings. The green sand molding conditions were about the same in the various plants. The characteristics of the green sand facings used are shown in Table II.

At various times throughout the investigation, test castings were poured in cored molds and combinations of core and green sand molds. No attempt has been made to determine the sand characteristics of the cored molds. Also, during these experiments, various mold preparations such as rubber compounds, alcohol, powdered aluminum, powdered magnesium, etc., were used. The results of these mold preparations are given later.

c. Forms of test castings. The various forms of test castings from which the test bars were made are shown in Figs. 1, 2 and 3. All of the test castings were poured as "detached" castings. Figs. 1 and 2 are of the usual type employed by foundrymen to obtain "attached" test castings for specification work. This test casting is not the same as the so-called "attached coupon bar." Fig. 3 shows a modified form of test casting which was used in the latter part of the investigation. This modification was used primarily to show how different surfaces of the same steel casting are affected by both core and green sand mold conditions.

d. Heat treatment. Most steel foundries now heat treat their castings for commercial use in order to obtain the higher mechanical properties which the trade demands. The test castings were pre-

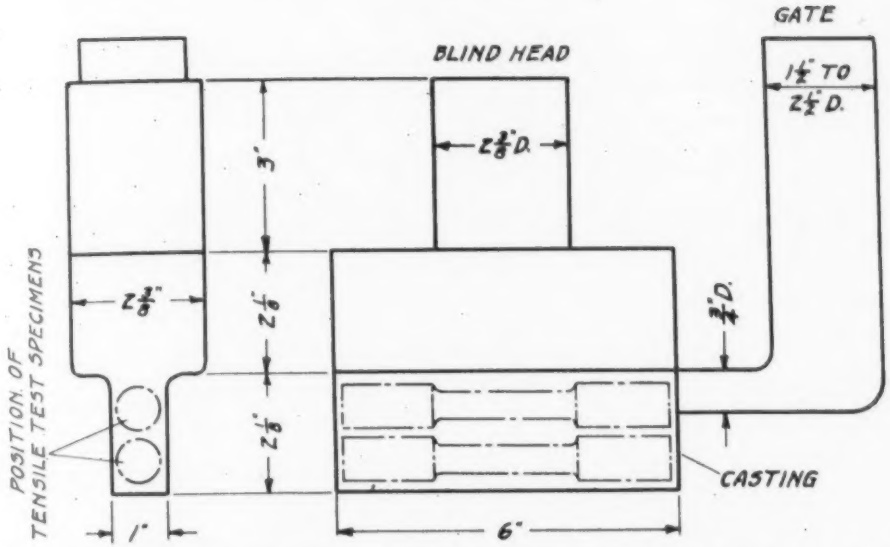


Fig. 1—Form of Test Casting Used to a Limited Extent by Foundry "A" from Which Tensile Bars Were Obtained.

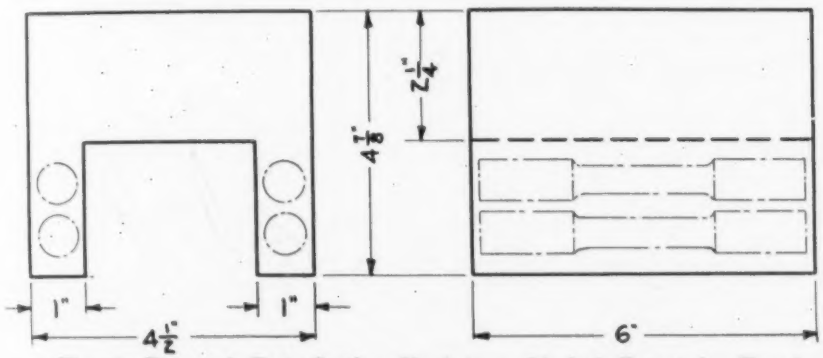


Fig. 2—Form of Test Casting Used to a Limited Extent by Foundry "B" from Which Tensile Bars Were Obtained.

viously heat treated at the various foundries where they were cast. These treatments are shown in Table I. In general, the heat treatment consisted of normalizing from 1600 to 1650 degrees Fahr. (870 to 900 degrees Cent.) or annealing from the same temperature range to a black heat. Various heat treating experiments were also conducted in the course of this work with the hope of improving the ductility of the steels which had been deoxidized with aluminum.

3. Test methods and equipment

The main test used for determining the mechanical properties of the test castings was the tensile test. All test bars were machined, after heat treating, from the test castings (Figs. 1, 2 and 3) and

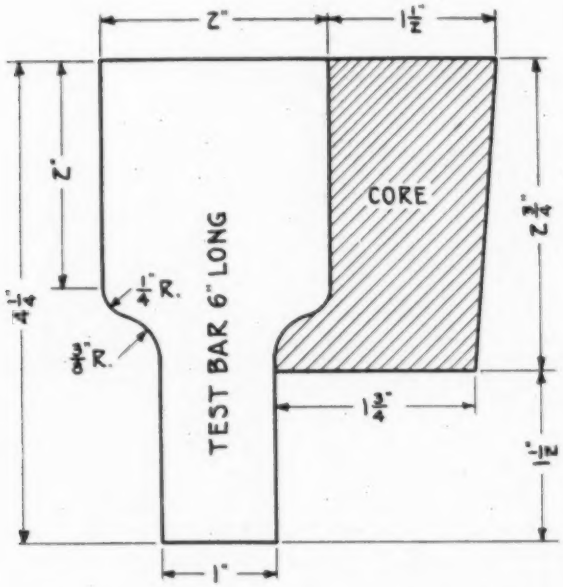


Fig. 3—Experimental Form of Test Casting Adopted by The Steel Castings Development Bureau for Tensile Bars to Show the Effects of Both Core and Green Sand on the Cast Steel.

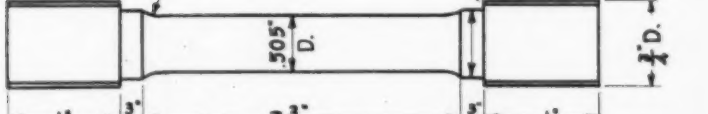


Fig. 4—Tensile Test Bar.

were of the form and dimensions shown in Fig. 4. The bars were broken on a 50,000-pound capacity Amsler testing machine. The yield point was determined from the automatically drawn stress-strain curves which were determined in each case.

The Izod impact test was used to a very limited extent in a preliminary way to supplement the ductility values as measured by the tensile test. Only 16 bars were tested. The results showed that as the ductility of the castings increased there also was a slight tendency toward higher resistance to impact. However, the Izod determinations on steel castings were not considered to be discriminating enough to be of importance in this work.

IV. Results

Most of the experimental test castings were made in the presence of the research associate, J. V. McCrae. The heats witnessed in this manner consisted of all of the heats at Company "A" and Heats No. 33-268-269-270 at Company "B."

1. *Effect of varying ordinary foundry conditions*

a. Average properties of green sand castings. No detailed data were available to show the chemical and physical properties and structural characteristics of the customary product in green sand castings, so a study was first made of 100 consecutive heats. The facilities of Company "A" were used for this work. These steels were melted, cast and normalized in accordance with what will be called "standard" practice. This consisted of charging about 20 per cent of boiler punchings, 15 per cent of light punchings from cold-rolled sheet, 25 per cent of heavy ball bearing punchings and the remainder, of return shop scrap (gates, heads, castings, etc.). The charge was melted with small quantities of iron ore, lime, ferrosilicon, etc., as required, and after about 1 hour and 15 minutes the metal was ready to cast. The heat was first tapped into a 2-ton teapot ladle where the ferromanganese was added. From this ladle, the metal was poured into the working ladles, the capacities of which varied from 70 to 1200 pounds of steel. All of the test castings were poured from the 70-pound ladle, two test castings being obtained from each ladle. One pound of aluminum per ton of steel was added in this ladle and the metal immediately poured into the test molds.

Except when otherwise indicated, all test castings made by this company were of the form and dimensions shown in Fig. 1. After normalizing, these were machined into tensile test bars. The customary normalizing practice used for these heats consisted of heating 2 hours at 1650 degrees Fahr. (900 degrees Cent.) followed by air-cooling. The results of the tests on the first 100 consecutive heats were tabulated together with the details of the melting practice. The 100 melts are grouped according to carbon contents in Fig. 5, the group variations being limited to 0.05 per cent carbon beginning at 0.13 per cent and ending at 0.33 per cent. Two additional groups are included consisting respectively, of steels containing less than

0.13 per cent carbon and those with 0.33 per cent or more. In Fig. 5 the different properties are plotted against the average carbon contents of these groups.

The data obtained show that 93 per cent of the test specimens in the group containing 0.13 to 0.18 per cent carbon had showed satisfactory tensile properties, whereas only 16 per cent in the group

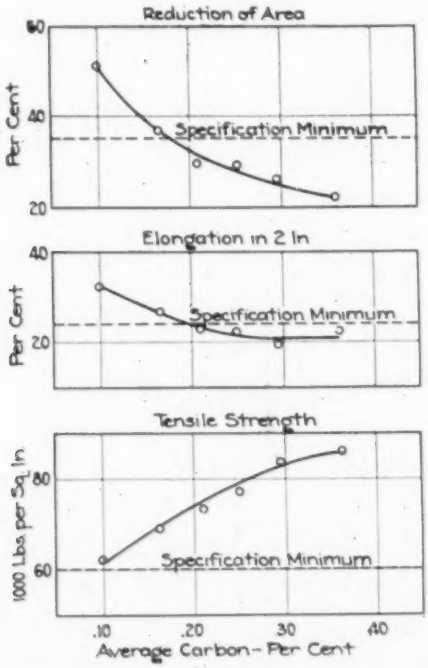


Fig. 5—Effect of Carbon Content on Physical Properties of Test Bars from 100 Consecutive Heats to Show the Variations Meeting Specifications in Plant Production.

of next higher carbon content passed requirements. With carbon above 0.28 per cent, none of the heats showed satisfactory ductility, less than 2 per cent of the test specimens had ductility properties within the desired limits. The majority of the 100 heats appeared to be "hard," i.e., the steels generally had higher tensile strength than was desired but were low in ductility. It is apparent from Fig. 5 that the carbon content must be kept at low values, if the majority of the heats produced under the so-called "standard" melting and normalizing practice outlined above are to meet the specification requirements.

The problem presented for solution is well illustrated in Table III. In this table the chemical and mechanical properties of twelve

Table III
Results of Chemical Analyses[1] of High and Low Ductility Steel Castings from 100 Consecutive Heats

Heat No.	Tensile Properties	Chemical Composition Per Cent C	Mn	Si[2]	P	S	Ni	Cr
4884	good	.29	.66	.50	.024	.044	.02	.02
4914	good	.19	.69	.32	.013	.046	.05	.02
4943	good	.25	.88	.52	.015	.042	.01	.02
4944	good	.22	.75	.46	.015	.048	.03	.03
4952	good	.25	.68	.30	.012	.052	.03	trace
4964	good	.19	.70	.36	.012	.050	.03	.02
	Average	.23+	.72	.41	.015	.047	.03	.02—
4896	poor	.21	.69	.29	.014	.047	.02	.01
4898	poor	.20	.79	.46	.018	.053	.04	.03
4904	poor	.24	.87	.58	.014	.046	.02	.03
4920	poor	.24	.68	.36	.014	.050	.06	.01
4929	poor	.22	.74	.40	.016	.048	.01	.02
4975	poor	.23	.71	.36	.018	.047	.02	.01
	Average	.22+	.75	.41—	.017	.049	.03—	.02—

	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Elongation in 2 Inches Per Cent	Reduction of Area Per Cent
6 good heats, average[3]	39,900	71,400	27.3	41.0
6 poor heats, average[3]	43,400	75,900	20.0	25.2

[1]All chemical analyses, unless stated otherwise by J. V. McCrae.
[2]Analysis made on test ingot and checked on tensile test bars.
[3]All bars given "standard" normalizing before test 1650 degrees Fahr. (900 degrees Cent.) 2 hours air cool.

heats selected from the 100 consecutive heats are summarized. Six of these were selected at random to represent heats which showed satisfactory ductility after the "standard" normalizing while the six others were selected at random to represent heats which showed unsatisfactory elongation and reduction of area under similar practice. The average chemical composition was practically identical in both cases insofar as carbon, manganese, phosphorus, sulphur, silicon, nickel and chromium were concerned. The average elongation and reduction of area, however, of the second set of six steels were very much lower than the corresponding properties of the six comparison steels.

b. Average properties of dry sand castings. Tensile tests were made on castings selected at random from 32 heats containing approximately 0.20 to 0.30 per cent carbon. The castings were not made especially for the tests, but were taken from heats made early in the course of this investigation by Company "B" who supplied test castings of the form shown in Fig. 2. The tensile properties together with the results of chemical analyses are summarized in Table IV. About one-fifth (18 per cent) of the specimens representative of the 32 heats made by Company "B" failed to meet the specifica-

tion requirements of 35 per cent or more reduction of area, and, in addition, about 5 per cent showed less than the required 24 per cent or more elongation in 2 inches. The majority of the test specimens showed typical silky cup fractures but about 12 per cent of the fractures contained small "bright spots." These were small areas of high lustre giving to the fractures the appearance which is sometimes referred to as "open-grain fractures."

It was thought that the superior tensile properties of the dry-sand castings as compared with the green sand castings might, in part, be the result of differences in the heat treating practice used by the different foundries. Hence, 12 test castings from Company "A" were submitted to the other foundry, Company "B," and an equal number from the latter were submitted to the first foundry for plant treatment. The treatment used by the first consisted of normalizing at 1650 degrees Fahr. (900 degrees Cent.) for 2 hours while in the second foundry the treatment was the same except that furnace cooling to a black heat was used instead of air cooling. The tests showed that the results from these two different treatments by the two different companies were practically the same.

c. Miscellaneous variables

1. Segregation. Steel castings do not, in general, show so great a degree of structural, chemical or physical homogeneity as wrought steels and the variations encountered may be of considerable magnitude. Duplicate and preferably triplicate determinations are advisable in most experiments if very wide conclusions are to be based upon the results.

An illustration of some of the difficulties encountered in interpreting the results of tests is given in Fig. 6. Fractured tensile test specimens from heats Nos. 4885 and 4891 which were originally reported to contain very nearly the same carbon but which showed widely different values of elongation and reduction of area were selected for microscopic examination. Study of the individual test specimens indicated a considerable difference in carbon content which was subsequently confirmed by chemical analysis. A check analysis showed 0.20 and 0.31 per cent carbon for the test bars, whereas the original values on the test ingots were 0.24 and 0.26 per cent respectively. Such variations in carbon content may account, in part, for the differences in ductility observed.

2. Design of test castings. Another factor considered relates to

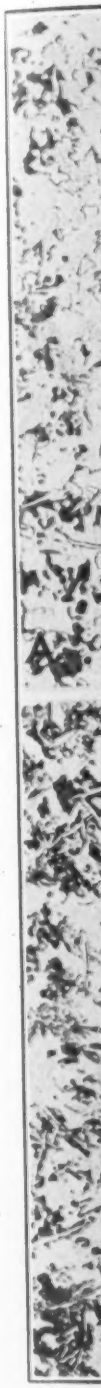


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Fig. 6—Characteristic Microstructures of Normalized Cast Steels Showing Widely Different Tensile Properties.

A and B—Heat 4891 Reported to Contain C 0.24, Mn 0.63, Si 0.53 but Found to Contain C 0.20. Tensile Strength, 73,000; elongation, 29.5%; reduction of area 46.0%. Normalized 1½ Hrs. at 1600 Degrees Fahr.

C and D—Heat 4885 Reported to Contain C 0.26, Mn 0.73, Si 0.48 but Found to Contain C 0.31. Tensile strength, 80,500; elongation, 18.0%; red. area, 20.5%. Normalized 2 Hrs. at 1645 Degrees Fahr. Etched in 5% Nital. A and C × 100; B and D × 500.

Table IV
Tensile Properties and Composition of Acid Electric Steel Test Castings Poured in Dry Sand Molds

Test castings were made according to regular plant practice.

Heat No.	Chemical Composition, Per Cent C	Mn	Si	S	P	Tensile Properties Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Elongation in 2 Inches Per Cent	Reduction of Area Per Cent	Remarks
Foundry "B"; Heat treatment, annealed for 6 hrs. 1600 to 1650°F. (870 to 900°C.) furnace cooled.										
31-128	.22	.65	.35	.048	.032	42,000	72,000	26.0	34.5	Bright spots
31-128						41,000	71,000	24.0	27.5	Bright spots
31-144	.20	.67	.35	.047	.033	40,000	66,500	31.0	38.0	Silky
31-144						40,000	66,000	30.0	38.0	Silky
31-157	.22	.65	.33	.048	.032	45,000	71,000	22.0	24.5	Silky
31-157						43,000	71,500	21.0	21.0	Silky
31-162	.23	.65	.35	.047	.033	39,500	65,000	34.5	51.0	Silky cup
31-162						38,500	64,500	37.0	53.5	Silky half cup
31-163	.22	.68	.33	.049	.032	37,500	67,000	25.0	25.0	Bright spots
31-163						37,500	66,500	25.0	37.0	Bright spots
31-165	.23	.64	.37	.047	.033	35,500	66,500	28.5	35.5	Silky
31-165						34,500	65,500	25.5	28.0	Bright spots
31-188	.23	.63	.35	.046	.032	36,000	70,000	28.0	46.5	Silky
31-188						37,500	70,000	27.5	48.0	Silky half cup
31-197	.22	.69	.37	.048	.031	38,000	64,500	33.0	53.0	Silky cup
31-197						37,500	64,000	34.0	55.5	Silky cup
31-201	.22	.61	.35	.045	.034	36,000	67,500	32.5	48.5	Silky cup
31-201						37,000	67,500	32.0	50.5	Silky cup
31-203	.19	.57	.33	.044	.035	36,500	63,500	29.0	34.0	Silky
31-203						37,000	63,500	32.5	38.0	Silky
31-204	.23	.65	.38	.045	.034	35,000	67,000	30.0	38.5	Silky
31-204						37,000	66,500	30.0	43.0	Silky half cup
31-205	.22	.60	.35	.045	.034	39,000	66,000	34.0	51.5	Silky cup
31-205						34,000	65,500	31.5	48.5	Silky part cup
31-208	.22	.68	.35	.045	.034	39,500	68,500	31.0	46.0	Silky half cup
31-208						38,500	68,000	32.0	45.0	Silky half cup
31-212	.20	.61	.35	.045	.034	40,500	63,500	33.0	48.0	Silky
31-212						40,000	63,500	28.5	38.5	Silky cup
31-213	.23	.65	.38	.046	.033	38,500	67,000	29.0	50.0	Silky cup
31-213						39,500	68,000	32.5	48.0	Silky half cup
31-214	.21	.59	.34	.046	.032	46,500	64,000	32.0	57.5	Silky cup
31-214										
31-216	.20	.55	.29	.045	.033	44,500	63,000	33.0	54.5	Silky half cup
31-216										
31-218	.22	.68	.42	.045	.034	40,500	67,000	36.5	48.5	Silky
31-218						38,500	67,500	29.5	35.0	Bright spots
31-221	.22	.61	.32	.047	.034	42,000	66,000	29.5	37.0	Silky
31-221						41,000	66,000	31.5	40.0	Silky
31-240	.22	.65	.34	.045	.034	38,000	66,500	33.0	48.5	Silky half cup
31-240						37,500	67,000	33.0	49.0	Silky part cup
31-243	.19	.63	.30	.045	.033	38,500	61,000	36.0	57.5	Silky cup
31-243						37,500	61,000	32.0	44.0	Silky. Slag inclusion at break.
31-246	.22	.71	.33	.047	.034	38,000	65,000	33.0	48.5	Silky cup
31-246										
31-248	.21	.67	.35	.045	.032	40,000	62,500	33.0	46.5	Silky half cup
31-248						39,000	61,000	28.5	37.0	Silky
31-250	.22	.70	.35	.047	.033	39,000	64,000	32.5	48.0	Silky
31-250						39,000	64,000	28.0	46.5	Silky
31-251	.19	.62	.31	.046	.034	34,500	63,000	36.5	53.0	Silky cup
31-251						35,500	63,000	34.5	53.5	Silky cup
31-253	.22	.72	.37	.046	.032	39,500	71,000	31.0	46.0	Silky
31-253						37,500	62,500	24.0	41.0	Silky
32-2	.22	.61	.31	.045	.030	37,500	67,000	30.0	36.5	Silky half cup
32-2						37,500	68,000	32.5	54.5	Silky half cup
32-3	.18	.58	.30	.045	.030	40,500	67,500	28.5	27.0	Silky
32-3						40,000	68,000	25.0	30.5	Silky
32-5	.22	.63	.33	.046	.030	38,000	65,000	33.0	51.0	Silky cup
32-5						38,000	65,500	32.5	46.0	Silky cup
32-6	.23	.65	.33	.045	.030	36,500	71,500	30.0	36.0	Silky
32-6						37,500	64,000	32.5	46.0	Small bright spots
32-11	.23	.74	.24	.044	.030	41,500	67,000	23.5	26.0	Silky
32-11						39,500	67,500	27.5	30.5	Silky
32-14	.22	.66	.35	.042	.030	35,000	65,000	32.5	50.5	Silky
32-14						36,000	65,000	33.5	50.5	

Table V

Comparative Tensile Properties of Test Castings of Different Forms

Form of Test Casting	Heat No.	Composition, Per Cent C	Mn	Si	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Elongation in 2 Inches Per Cent	Reduction of Area Per Cent
Regular (Fig. 1)	5301	.21	.75	.25	47,500	71,000	27.0	36.0
					42,000	69,000	27.0	34.0
1" Round		.21	.75	.25	43,000	69,500	27.0	34.5
					47,000	69,500	27.0	34.5
Regular (Fig. 1)	5284	.23	.76	.39	52,500	78,000	18.5	22.5
					50,500	76,000	15.5	23.0
1" Round		.23	.76	.39	49,000	75,500	20.0	28.0
					48,000	75,500	20.0	25.5

the form of test casting. The one regularly used (Fig. 1) had sharp corners in the portions from which the tensile specimens are machined. Such corners affect the process of dendritic formation and may introduce a line of weakness extending into the test bar.

Accordingly, two heats were cast into rounded sections as well as into castings of the form illustrated in Fig. 1. The same heads and gates were used in both cases but the casting proper was about 1-inch round in place of the rectangular section, and was poured into a core instead of in the green sand used in regular practice. The results of tensile tests, after the customary normalizing treatment, are summarized in Table V. It will be observed that these changes in section and mold practice did not produce noticeable differences in the ductility.

3. Heat treatment. The experiments previously described indicated the need for improvements in tensile properties, but did not throw light upon the causes of the difficulties or possible remedies. The low ductility of the majority of heats might originate from one or both of two sources, namely, the heat treatment or the foundry practice.

The heat treatment cycle used for the previous tests was relatively short and might reasonably have contributed to the "hardness" and low ductility of the majority of the first 100 heats. Therefore, the heat treatment cycle was first investigated.

Thermal analyses were made of two of the cast steels since these normally contain somewhat higher silicon than commercial wrought steels of similar carbon contents. The Ac_3 transformation temperature was considerably higher in both of the cast steels than in the low-silicon steels usually employed for rolled and forged prod-

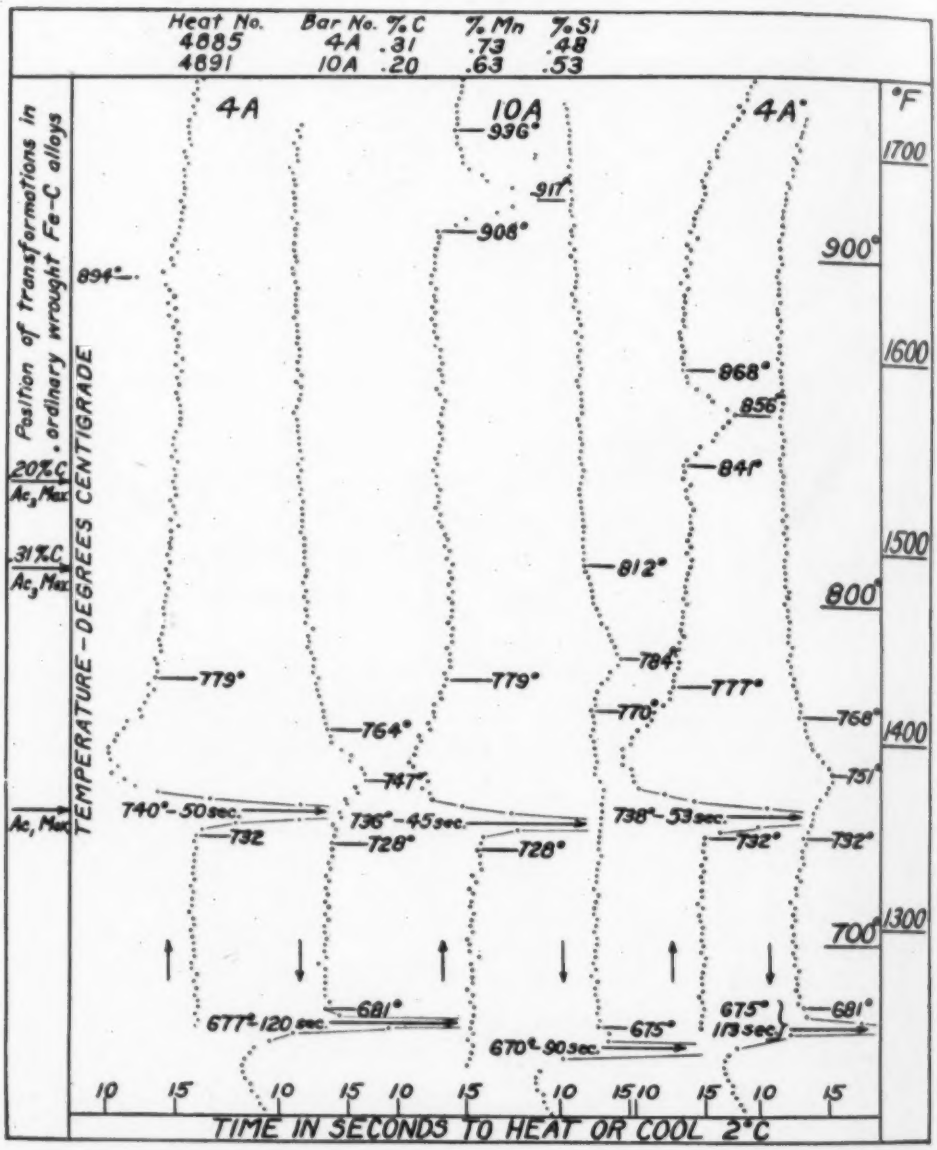


Fig. 7—Inverse Rate Heating and Cooling Curves on Specimens of Cast Steel Showing Widely Different Tensile Properties after Normalizing.

ucts. (Fig. 7). On this account the first modifications made in the "standard" normalizing cycle were to increase the temperature and decrease the rate of cooling. The results obtained are given in Table VI.

It is known that steels containing like proportions of carbon, manganese, silicon, etc., frequently react differently in heat treatment. Such differences in heat treatment properties are probably associated

Table VI
Effect of Varying Heat Treatments on the Tensile Properties of Cast Steels
Heat Treatments were Carried Out on the Test Castings

Heat No.	How Classed under "Standard" Normalizing*	Composition Per Cent C	Mn	Si	Heat Treatment[1]	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
					Higher Initial Temperatures of Heating				
4922	g	.12	.58	.28	1650°F—2 hrs.—air cool	39,000	62,000	32	51.0
						37,500	56,500	34	43.5
					1800°F—3 hrs.—fur. cool	38,500	57,000	34	43.0
						46,000	66,500	26	37.5
4889	g	.27	.65	.50	1650°F—2 hrs.—air cool	44,400	74,800	24.5	40.0
						44,000	73,500	24	26.5
					1700°F—3 hrs.—fur. cool	39,000	69,500	27	36.5
						41,500	71,000	27.5	40.5
4924	p	.15	.71	.31	1650°F—2 hrs.—air cool	41,200	73,700	26	34.0
						43,500	69,500	23.5	28.5
					1800°F—3 hrs.—fur. cool	43,000	70,000	23.5	30
						38,500	68,500	25.0	27
4981	p	.26	.64	.40	1650°F—2 hrs.—air cool	42,000	75,500	21	24
						40,500	72,000	26	30
					1700°F—3 hrs.—fur. cool	41,500	74,000	27	35
						42,500	74,500	27	36.5
					Water Quenching and Tempering				
4922	g	.12	.58	.28	1650°F—2 hrs.—air cool	39,000	62,000	32	51
						40,000	60,500	36.5	53.5
					1750°F—2 hrs.—water	50,500	70,000	25	41
					1200°F—2 hrs.—air cool	46,500	69,500	29	44
4889	g	.27	.65	.50	1650°F—2 hrs.—air cool	44,400	74,800	24.5	40.0
						55,500	74,500	23.5	36.5
					1650°F—2 hrs.—water	49,000	83,500	23.5	36.5
					1200°F—2 hrs.—air cool	53,500	83,500	25	39.0
4924	p	.15	.71	.31	1650°F—2 hrs.—air cool	41,200	73,700	26	34
						58,000	83,500	22.5	38.5
					1750°F—2 hrs.—water	50,500	80,000	15.0	27.0
					1200°F—2 hrs.—air cool	56,500	81,500	19.0	27.0
4981	p	.26	.64	.40	1650°F—2 hrs.—air cool	42,000	75,500	21	24
						64,000	88,500	13.5	22
					1650°F—2 hrs.—water	62,000	87,500	22.0	28.5
					1200°F—2 hrs.—air cool	59,000	87,500	20.0	34.5
					Repeated Normalizing				
4979	p	.21	.62	.42	As cast	33,500	73,000	17.5	9.5
					1650°F—2 hrs.—air cool[2]	47,000	77,000	23.0	27.5
					ditto	50,000	80,000	23.0	27.5
					Long Time at 1625°F				
4898	p	.20	.67	.46	1650°F—2 hrs.—air cool[2]	42,200	81,000	21.5	26.5
					1625°F—15 hrs.—air cool	54,000	82,000	23	33.5
						51,500	83,600	19	25.5
4975	p	.20	.55	.36	1650°F—2 hrs.—air cool[2]	42,200	73,800	19	23.5
					1625°F—15 hrs.—air cool	46,000	72,000	23	29
						48,000	72,000	22	29
4914	g	.18	.74	.32	1650°F—2 hrs.—air cool[2]	39,400	68,800	30	40.5
					1625°F—15 hrs.—air cool	47,000	68,000	19	25.5
						45,000	65,000	30	46.5
4964	g	.21	.64	.36	1650°F—2 hrs.—air cool[2]	37,500	64,200	30	46.5
					1625°F—15 hrs.—air cool	44,000	64,000	26	33
						41,500	74,000	26	35

[1]All heat treatments except where stated otherwise were carried out in the laboratory.
[2]Heat treatment carried out in the plant under commercial conditions.
*g=good p=poor
Remarks—Average 2 tests treated in laboratory furnace.

with various causes, but in at least some cases are believed to be due to differences in the character and distribution of "nonmetallics."

When finely dispersed these may offer centers for easy reprecipitation and so make difficult the removal of the original dendrites in cast steels. Quenching is more effective in preventing precipitation on these nuclei than is slow cooling. Therefore, samples from heats which showed good tensile properties under "standard" normalizing and those which showed relatively poor tensile properties were

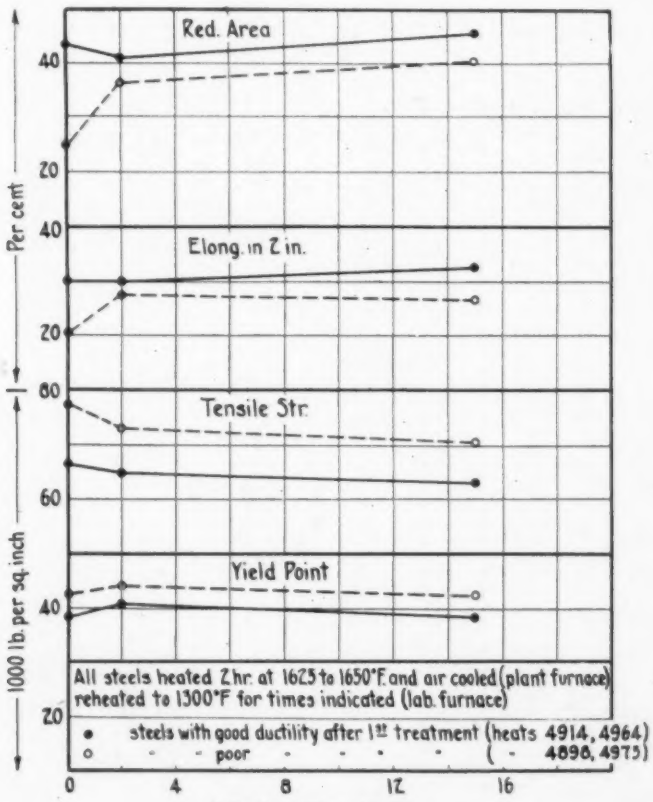


Fig. 8—Effect of Time of Reheating at 1300 Degrees Fahr. on the Tensile Properties of Cast Steels Poured in Green Sand Molds.

quenched and subsequently tempered at 1200 degrees Fahr. (650 degrees Cent.). Prolonged soaking at high temperatures is helpful in breaking up dendritic segregation. The results of the various tests are included in Table VI.

Long heating at temperatures just under or in the transformation range has frequently been found to increase ductility. This was also tried and the results are shown in Fig. 8.

Of the five variables in heat treatment investigated only one

prolonged tempering at 1300 degrees Fahr. (705 degrees Cent.) produced quite definite and appreciable improvement in the ductility of the steels. Increase in the temperature from which the steel was cooled, combined with a slower cooling rate (furnace cooling), was not effective in increasing the ductility of the samples. Water quenching followed by tempering at 1200 degrees Fahr. (650 degrees Cent.) did not bring the poor heats within the specification requirements although either a higher tempering temperature or a longer time of tempering might have produced a sufficient improvement so that most of the steels would have passed the ductility requirements. (Table VI). The use of water quenching, in general, followed by tempering would not appear to be practicable on account of the intricate design of the average foundry product. The results shown in Table VI for heat 4979 indicate that repeated heating to 1650 degrees Fahr. (900 degrees Cent.) followed by air cooling does not offer much, if any, advantage over a single treatment. Likewise, prolonged heating at 1625 degrees Fahr. did not improve the ductility except in one heat (No. 4975) where a slight increase was observed. Individual test specimens of the other heats showed low ductility values as measured by both elongation and reduction of area after the prolonged heating. It is probable that the heating was not carried above the transformation temperature range in either of these treatments because Fig. 7 shows that the Ac_3 range is higher in these cast steels than in corresponding steels low in silicon.

The one type of treatment which effectively raised the ductility was a tempering at 1300 degrees Fahr. (705 degrees Cent.) subsequent to normalizing. As shown in Fig. 8, there was no marked change in ductility produced by tempering those heats which showed high elongation and reduction of area after simple normalizing but the so-called poor heats showed a relatively large increase in both reduction of area and elongation when tempered for only two hours at 1300 degrees Fahr. (705 degrees Cent.). Longer tempering produced further but small increases in ductility.

A 15-hour heating at 1300 degrees Fahr. (705 degrees Cent.) produced a considerable degree of spheroidization of the cementite which was accompanied by an increase in the elongation and reduction of area, although there was no appreciable change in the dendrites which were not removed by the simple normalizing. Fig. 9 shows a comparison of the microstructure of typical test castings

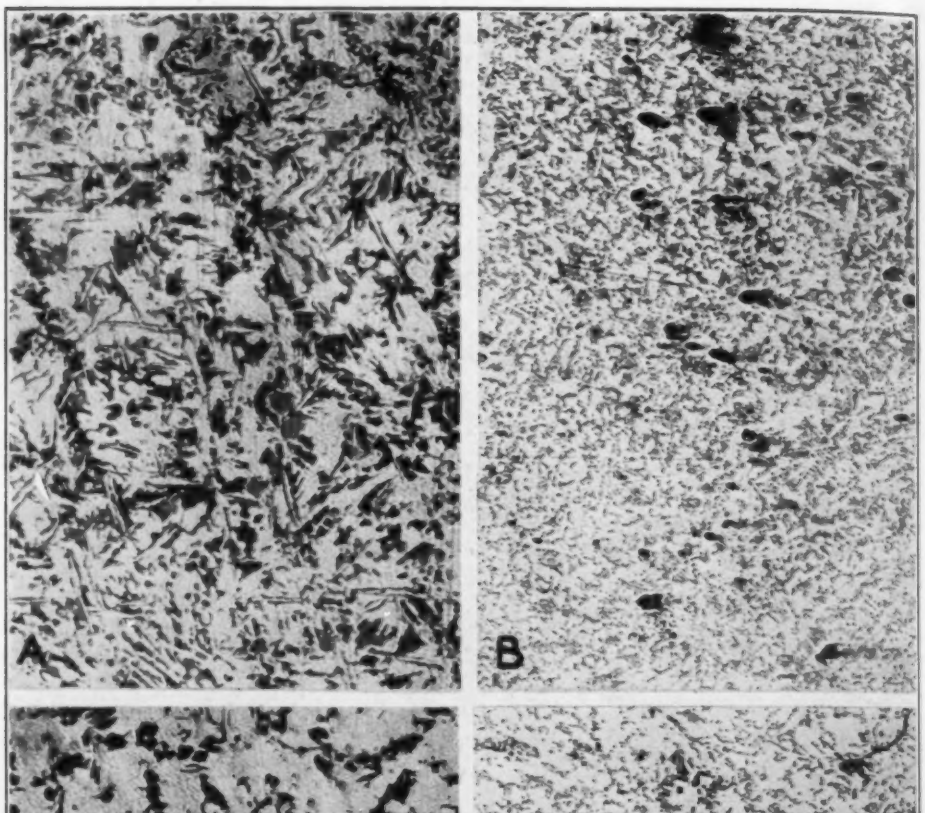


Fig. 9—Effect of Time of Reheating at 1300 Degrees Fahr. on the Microstructures of Two Cast Steels which Previously Showed Unsatisfactory Ductility after their Normalizing Treatments. A—Specimen from Heat 4898, Normalized 1650 Degrees Fahr. 2 Hrs. B—Same as A but Followed by Reheating for 15 Hours at 1300 Degrees Fahr. C—Specimen from Heat 4975, Normalized Same as A. D—Specimen from Heat 4975, Same Treatment as B. Test Castings Were Poured in Green Sand Molds. Etchant 5% Picric Acid. × 100.

Table VII
Effect of Character of Scrap on Tensile Properties of Cast Steels

Heat No.	Charge in Per Cent			Composition Per Cent			Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
	Crop Ends	Axles	Return Shop Scrap	C	Mn	Si				
5118	100	...	...	.30	.62	.36	55,500	82,000	20	25.5
							55,000	81,500	broke in machine marks	
5119	100	...	...	.30	.79	.31	51,000	80,000	23	23.5
							50,000	77,500	23.5	26.0
5126	47.5	2.5	50	.24	.64	.52	47,500	74,000	27.5	37.5
							47,500	72,000	27.0	34.0
5133	...	...	100	.26	.70	.44	43,000	78,500	21.5	27.0
							43,000	78,500	22.0	26.5
5137	...	...	100	.24	.70	.39	46,000	71,000	23.5	26.0
							46,500	71,500	24.5	30.0
5138	...	...	100	.23	.66	.38	43,000	69,000	28	34.5
							43,000	69,000	31	40.0

poured in green sand molds with steel deoxidized with aluminum in the condition as normalized and as reheated to 1300 degrees Fahr. (705 degrees Cent.) for fifteen hours and air cooled.

4. Casting temperature. The results obtained suggest that there is a tendency toward higher tensile properties if the castings are poured as hot as practicable. This point is further illustrated by Fig. 10, which represents three test castings poured at rather widely varying temperatures from the same shank ladle of metal. The results shown in this figure are typical of two tests of this kind. In each case, the heat was tapped "very hot" and the first test casting poured as quickly as possible, the second casting was poured in the normal manner, while the third was poured after the metal in the ladle had cooled to what was thought to be the lowest practicable casting temperature. It should be noted that the results from this single heat, picked at random, are consistent with the general indications of the 100 consecutive heats previously reported.

5. Character of scrap used in the charge. As previously indicated the largest part of the charge used in the production of the test castings ordinarily consisted of rather small scrap. Even though such is fairly clean and free from rust, it will oxidize during melting to a greater degree than heavy scrap. Several heats were made in which the character of the scrap was varied in order to determine whether this factor bears any important relation to the ductility of the castings. The results are summarized in Table VII.

Unfortunately, two of the six heats, in which only heavy scrap was used, contained relatively high carbon which by itself would

Table VIII
Effect of Alloying with Either Nickel or Vanadium on the Tensile Properties of Green Sand Test Castings[1]

Heat No.	Chemical Composition Per Cent C	Mn	Si	Ni[2]	Other Elements	Tensile Properties Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
Ave. of 32 heats	.18 to .23			none	none		73,000	23	29
5213	.16	.57	.26	.46	....	41,000	63,500	32.0	42.0
						41,200	63,000	35.0	45.0
						39,000	63,000	32.5	47.0
5239	.20	.63	.29	1.14	Cr .03	48,500	74,000	23.5	34.0
						50,500	74,000	24.0	30.5
						47,500	74,000	25.0	28.0
5301	.21	.75	.25	none	....	47,500	71,000	27.0	36.0
						42,000	69,000	27.0	34.0
5301	.21	.75	.25	.32	....	43,000	69,500	29.0	39.0
						42,000	68,000	24.0	32.0
5301	.21	.75	.25	.56	....	44,000	70,000	27.0	34.0
						44,000	70,500	25.0	29.0
5301	.21	.75	.25	.84	....	49,000	73,500	24.0	27.0
						50,000	73,500	22.5	28.5
5337	.18	.53	.39	.48	....	50,100	71,000	17.5	25.0
5339	.19	.76	.37	.61	....	48,000	71,000	13.5	23.0
						47,000	72,000	13.5	23.0
5343	.16	.60	.32	.75	....	42,000	69,000	32.5	54.0
						42,000	71,000	32.5	54.0
5274[3]	.18	.73	.28	none	V .25	53,000	77,000	29.0	52.0
							porous bar for duplicate		
					V .25	63,500	85,500	11.0	16.5
						66,000	85,000	10.5	7.0

[1]In all cases except Heat 5274, 1 pound of aluminum per ton of metal was added to the shank ladle.
[2]Added in the 70-pound working ladle except for 5213 and 5239 in which it was added in the 300-pound ladle.
[3]Vanadium added as ferrovanadium, 35 per cent V.

tend to reduce the ductility. The results indicated that the ductility of test castings poured in green sand (one pound of aluminum per ton of metal added to the shank ladle before casting) was somewhat lower when light scrap was used in the charge rather than heavy scrap. In general, however, the question of the character of scrap used appears to play only a small part in the matter.

6. Alloying with either nickel or vanadium. It was believed that nickel additions in small amounts might have a beneficial effect on the ductility. The results of the low alloy heats are shown in Table VIII. Two of the heats, 5343 and 5213, containing about ½ and ¾ per cent of nickel respectively, showed a combination of good strength and ductility, but it does not appear justifiable to attribute these properties solely to the nickel. Several of the other heats containing nickel, for example, No. 5339, showed very low ductility. The results shown by heat 5301 without nickel further indicate that nickel additions are of less importance than those factors which are pri-

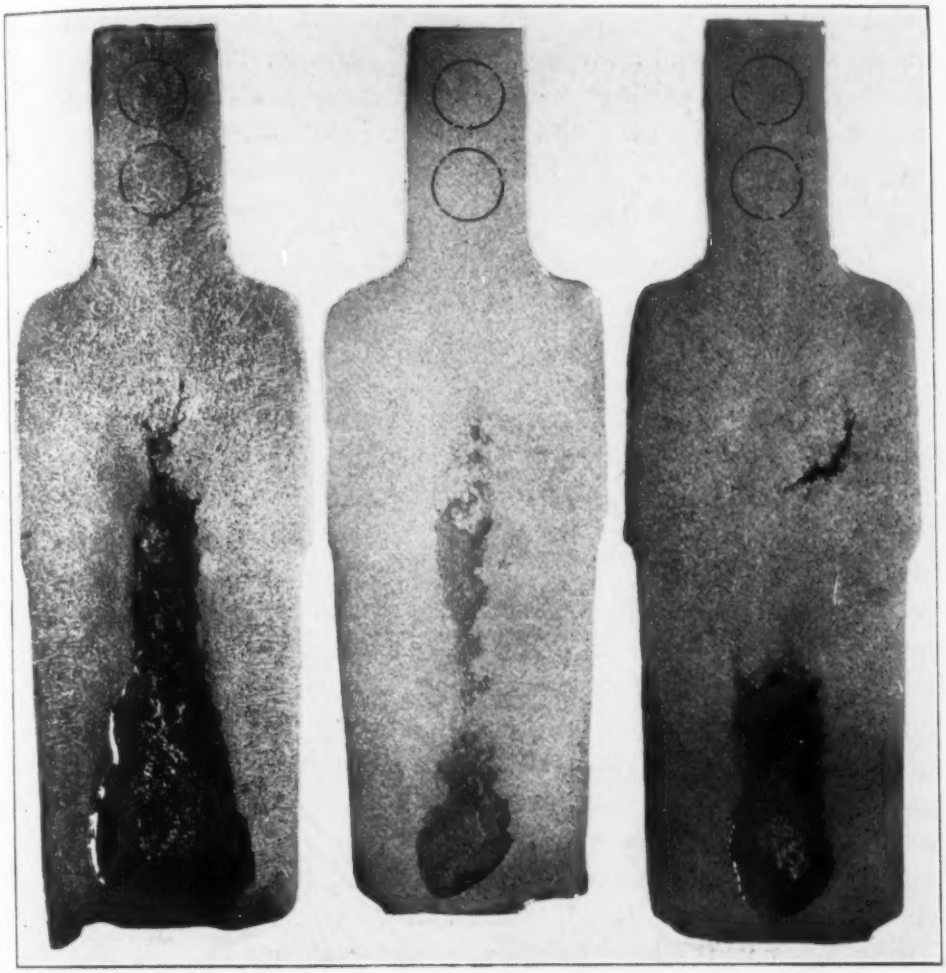


Fig. 10—Macrostructures of Three Test Castings Poured at Different Temperatures from Heat 5275.

A (Left)—Cast "hot," Tensile Strength, 78,500; Elongation, 21.5%; Per Cent Red. of Area, 23.5%. B (Center)—Cast at Intermediate Temperature, Tensile Strength, lbs. per sq. in., 75,250; Per Cent Elongation, 16.5; Per Cent Reduction of Area, 21.0. C (Right)—Cast "Cold," Tensile Strength, 76,800; Elongation, 14.5%; Red. Area, 19.0%. Test Castings Poured in Green Sand Molds; Etched With Ammonium Persulphate Solution. Reduced about ½ in Printing.

marily responsible for either high or low ductility obtained by the usual practice. Vanadium was added to a 70-pound ladle of metal from heat 5274. This ladle of metal was divided, aluminum being added to one part but not to the other.

One test casting containing 0.25 per cent vanadium without aluminum, showed good properties, but the corresponding bar was porous. The test castings to which aluminum had been added, in addition to vanadium, in the attempt to insure soundness in the green sand mold, showed exceptional low ductility.

From the above experiments, it seems that the additions of low amounts of the usual alloy elements will not appreciably improve the ductility of steel castings made in green sand, if, in addition, the steel is deoxidized with aluminum in the usual manner.

2. *Effect of various deoxidizers and deoxidation methods*

It is well known that the method of deoxidation or "degasification" in all steel making practice is of primary importance in determining the final properties of the steel. A number of experiments were made to show the effect of different so-called deoxidizers on the tensile properties of steel castings when added in various ways and in different amounts. On account of the widespread use of aluminum in steel casting practice, attention was first given to the use of this deoxidizer. Later, experiments were made with other less familiar "deoxidizers." These tests should be regarded as "pilot tests"; none of them were extensive enough to make definite recommendations concerning the use of any particular deoxidizer. In most cases, however, the general behavior of the "addition" was rather clearly indicated.

a. Aluminum. The use of aluminum in steel foundry practice as an addition to the steel before pouring into the molds is a precaution rather than an absolute necessity. In a considerable number of cases in this investigation, sound castings were obtained when no aluminum was added. Indeed, the claim has been made, that in normal foundry practice, sound castings can be obtained without the use of aluminum for about 80 per cent of the time.

In obtaining the results on the 100 consecutive heats, deoxidation was accomplished by the addition of one pound of aluminum per ton of steel in the shank ladle (70 pounds capacity) just before casting. Twenty-nine additional heats were made by the same company in which the same amount of aluminum was added to the steel but the addition was made to the metal in the large ladle during tapping. The melting practice followed in making these additional heats was practically the same as that followed in making the 100 consecutive heats.

The tensile properties of the test castings obtained are shown in Table IX. A comparison of these results with those obtained on the 100 heats shows that, while the ductility values were somewhat low in a number of cases, they were not so low as the values

Table IX

Tensile Properties of Test Castings of Steel Deoxidized by the Addition of 1 Pound Aluminum per Ton of Steel in the 2-ton Ladle

Castings were made in green-sand molds and heat treated by normalizing [1650°F (900°C), 2 hrs., air cooled]—by Co. "A"

Specimen Number	Chemical Composition, Per Cent C	Mn	Si	Tensile Properties Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
101	.21	.72	.44	49,500	73,000	25.0	29.5*
101A				49,500	78,000	16.5	27.0*
102	.51	.81	.46	59,500	106,500	18.5	26.5
102A				65,000	109,000	12.0	15.0*
103	.38	.79	.42	59,000	91,500	22.5	29.0
103A				60,000	92,500	24.0	23.0
104	.37	.71	.41	72,000	86,500	23.0	30.5
104A				55,500	87,500	19.0	22.5
105	.34	.77	.46	56,500	87,000	19.0	25.0
105A				55,000	86,000	18.0	21.0
106	.32	.53	.32	46,000	80,000	21.0	26.0
106A				47,000	79,000	18.0	22.5
107	.30	.97	.57	58,000	85,000	18.0	22.0
107A				55,000	87,000	23.0	30.0
108	.27	.66	.39	47,500	79,500	29.0	44.5
108A				47,500	79,500	23.0	23.0*
109	.20	.56	.26	45,500	72,000	16.0	23.0*
109A				42,500	62,500	9.0	18.0*
110	.41	.67	.44	44,000	70,000	22.5	35.0*
110A				44,000	70,000	15.0	21.0*
111	.17	.54	.24	39,500	65,500	19.0	29.0
111A				47,000	66,000	18.0	28.5*
113	.21	.72	.32	47,000	72,000	25.0	34.0
113A				46,000	71,000	27.5	34.0
114	.21	.63	.26	45,000	73,500	33.0	51.0
114A				44,500	72,500	15.0	24.5*
115	.22	.70	.36	44,500	70,500	22.5	27.0
115A				44,500	67,000	25.0	32.5
116	.23	.73	.39	44,500	69,000	25.5	29.5
116A				44,000	69,000	28.0	32.0
117	.20	.73	.38	44,000	70,000	32.5	49.0
117A				43,500	71,500	26.0	36.5
118	.12	.65	.35	48,500	72,500	26.0	31.0*
118A				43,500	68,500	32.0	46.0
119	.16	.68	.40	41,500	68,500	18.0	32.0*
119A				42,000	65,500	14.0	31.0*
120	.18	.69	.23	44,000	68,500	17.5	27.0*
120A				45,000	66,000	18.0	31.0*
121	.12	.50	.30	39,000	63,500	38.5	60.0
121A				39,500	64,000	34.0	57.0
122	.14	.52	.75	39,500	60,500	38.0	63.0
122A				39,000	62,000	36.0	61.5
123	.16	.74	.41	47,000	67,000	27.0	34.0
123A				45,500	65,000	26.0	35.5
124	.22	.73	.40	49,500	68,000	20.0	29.5
124A				43,000	74,500	28.0	35.5
125	.16	.69	.37	42,500	65,000	28.0	40.0
125A				43,000	65,000	27.5	34.5
126	.22	68	33	46,500	81,000	21.0	28.0
126A				47,000	78,500	21.0	24.0
127	.23	.69	.38	44,000	71,500	25.0	31.5
127A				42,500	71,000	23.0	27.0
128	.20	.66	.30	39,500	61,000	38.0	60.0
128A				40,000	61,000	36.5	63.5
129	.16	.50	.40	46,000	60,000	7.0	17.5*
129A				43,000	60,000	7.0	14.0*
130	.21	.68	.41	40,500	60,000	7.5	3.5*
130A				40,000	52,500	5.0	6.0*

*Porous bar.

Table X
Effect of Varying the Method of Aluminum Additions on the Tensile Properties of Low Carbon Cast Steels

Heat No.	Chemical Composition Per Cent C	Mn	Si	Aluminum Added Amount	When	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
5301	.21	.75	.25	1 lb. per ton	70 lb. ladle	47,500	71,000	27.0	36.0
						42,000	69,000	27.0	34.0
5301	.21	.75	.25	none	.....	39,000	72,000	33.0	49.0
						42,000	72,000	34.0	54.5
5392	.23	.74	.34	1 lb. per ton	2 ton ladle	44,000	74,000	20.5	25.0
5343	.16	.60	.32	1 lb. per ton	2 ton ladle	42,000	71,000	32.5	54.0
						42,000	69,000	32.5	54.0
5346	.20	.70	.29	1 lb. per ton	2 ton ladle	41,500	72,000	32.5	56.0
5347	.18	.66	.38	[1]	2 ton ladle	45,500	73,500	27.0	31.0
5348	.20	.65	.26	1 lb. per ton	2 ton ladle	42,500	73,000	31.0	50.0
						41,000	73,500	17.5	17.5
5349	.26	.77	.37	1 lb. per ton	2 ton ladle	43,000	76,500	18.0	25.0
						46,000	77,000	18.0	24.5
5350	.32	.75	.34	1 lb. per ton	2 ton ladle	49,000	79,000	17.0	22.0
						49,500	79,000	17.5	14.5

[1]More than the customary amount was added, the exact amount not known.

obtained with the 100 heats. It would seem that the products of deoxidation have had a better chance to rise to the surface and perhaps free the steel more from colloidal particles which would affect the ductility. Many of these bars, however, contained blowholes, thus showing that the steels had not completely "degasified" or that bad mold conditions had actually "regasified" the metal which did not contain sufficient deoxidizer to allow for the gases evolved by the mold. There is indirect evidence that the greater these mold gases the larger will be the amount of aluminum necessary to insure soundness and the lower will be the ductility of the steel.

The variation in the properties of cast steel to which aluminum has been added is further illustrated by the results in Table X. Aluminum was added in the small working ladle (70 pounds capacity) in one case and in the 2-ton ladle in others. It will be noted that for the particular heat of steel used, the ductility of the untreated steel was considerably higher than of the same steel treated with aluminum. In two cases, the ductility of castings made from steel deoxidized in the large ladle was considerably better than that of the steel deoxidized in the small ladle just before pouring and equal to the high value shown by the untreated steel. In other cases, however, the steel deoxidized in the large ladle was inferior in its ductility to that of the comparison heats. As these castings were poured from shank ladles, taken from the top portion of the large ladles, very soon after the steel had been "deoxidized" with

aluminum, it is probable that the low ductility encountered with some of the heats was due to inclusions of the products of "deoxidation".

In general, "pin holes" were prevalent in the majority of the castings in which the practice was followed of adding the aluminum in the large ladle. Although no definite conclusions are justified on the basis of these results alone, it is apparent that the problem

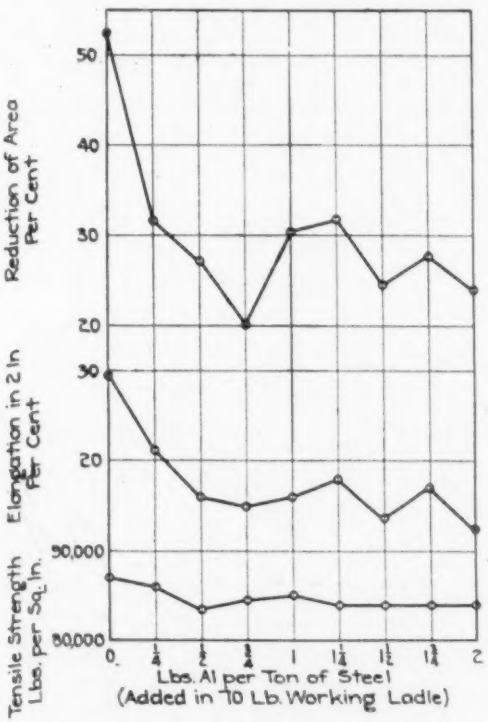


Fig. 11—Effect of Quantity of Aluminum Used as a "Deoxidizer" Immediately Before Casting on the Physical Properties of Steel Test Castings Made in Green Sand Molds. Heat 5680: Analysis C 0.26, Mn 0.63, Si 0.37 Per Cent. Normalized at 1650 Degrees Fahr. 2 Hrs. Each Point Represents Average of Two Tests.

is not one that depends entirely upon the time when the aluminum additions are made with respect to the time of casting. Evidently the initial condition of the steel plays an important part. In this respect, they tended to duplicate the condition which so frequently obtains in castings made from steel which has not been treated with aluminum. The porosity may have resulted from an insufficient amount of residual deoxidizer to compensate for the gases formed from the moisture of the mold.

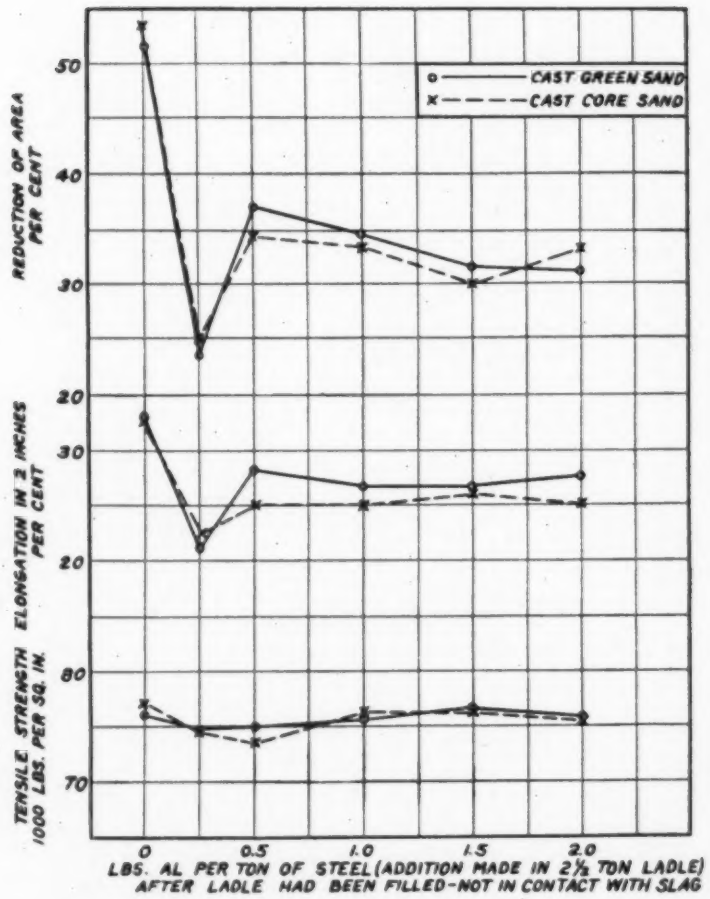


Fig. 12—Effect of Quantity of Aluminum Used as a "Deoxidizer" in 2½-Ton Ladle on the Physical Properties of Steel Test Castings Poured in Both Green and Core Sand Molds. Heat 33-268 (Foundry "B"). Analysis: C 0.24, Mn 0.70, Si 0.41 Per Cent. Normalized at 1650 Degrees Fahr. 2 Hrs. Each Point Represents Average of Two Tests.

The results of tests carried out for the purpose of studying the effect of varying the amount of aluminum added to green sand castings are given in Figs. 11 and 12. Two corresponding series of tests were carried out, one in the plant of Company "A", the other at Company "B". In the first series the amount of aluminum added was progressively increased up to 2 pounds per ton of steel, the additions being made in the shank ladle (70 pounds capacity). In the companion series, corresponding additions were used but were made in the large (2½ ton) ladle, the aluminum being added by fastening it to the end of a steel stirring rod and holding it beneath the surface of the metal. The test castings in this case were poured within approximately half a minute from a 150-pound capacity ladle

Table XI

Chemical Compositions of the Al-Si-Fe Alloys Used as Deoxidizers

Designation	Chemical Composition, Per Cent Al	Fe	Si	Actual[3] Addition Grams
Commercial ½" round Al rod	approx. 98.5			
Alsifer[1]	20	40	40	80
Al-Si No. 1[2]	32.0	39.2	30.1	50
Al-Si No. 2[2]	29.3	37.2	35.0	55
Al-Si No. 3[2]	6.7	51.9	40.9	230

[1]Approximate composition as given by manufacturer, Vanadium Corporation of America, 120 Broadway, New York.
[2]Analysis furnished by Bureau of Mines.
[3]Equivalent to 1 pound aluminum per ton of steel.

which had been filled by pouring over the lip from the 2½-ton ladle.

It will be noted in both Figs. 11 and 12 that the tensile properties corresponding to zero addition of aluminum were noticeably superior to those of any of the steels of the same series containing aluminum. As already mentioned, however, such values are not readily attainable; they are to be regarded somewhat as "ideals" or standards for comparison purposes rather than as results representative of general foundry practice.

As is evident from both series of tests, a relatively small addition of aluminum to the cast steel resulted in a marked change in the ductility properties of the steel. The decrease in the ductility does not bear a linear relationship to the amount of aluminum added. Both series of tests indicated a "ductility minimum" corresponding to a relatively early stage in the series of aluminum additions. In the tests in which the aluminum was added to the large (2½ ton) ladle this minimum corresponded to an aluminum addition of 0.25 pounds per ton whereas in the companion series in which the aluminum was added in the small working ladle, the minimum corresponded to an addition of 0.75 pounds per ton. However, since the two series of steels were made in different plants and in all probability differed in other respects, it cannot be stated with certainty that the difference in the method of adding the aluminum was wholly responsible for this observed difference in the "ductility minimums."

The results of both series of tests were in good agreement in showing that the effect of the progressive aluminum additions upon the tensile strength of the steel was relatively very much less than upon the ductility.

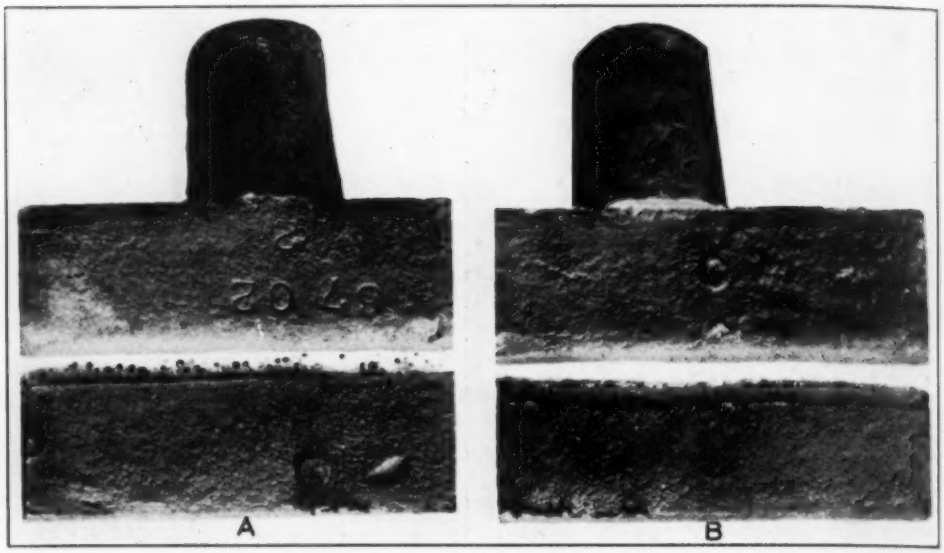


Fig. 13—Surface Appearance of the Opposite Faces of a Steel Test Casting Made in the Composite Mold of Core Sand and Green Sand, × ½. Reduced about ⅓ in Printing. Heat 6702, No Addition of "Deoxidizers" Were Made.
A—Green Sand Face; Note the Numerous Holes in the Metal Along the Fillet. B—Core Sand Face; Note the Sound Metal Along the Fillet.

In the tests in which the aluminum was added in the large ladle, test castings were poured in both green sand and core sand. No important differences or significant differences were obtained in the properties of the two sets of test castings prepared in this way (Fig. 12).

b. Special "deoxidizers". In order to determine whether sufficient deoxidation could be secured without lowering the ductility of the steel if the aluminum were in combination, tests with a number of special deoxidizers were carried out. Compounds of aluminum, silicon and iron were first used. One of these, "Alsifer", is a commercial product. Three others were prepared for the tests at the Pittsburgh Station of the Bureau of Mines, under the supervision of Dr. C. H. Herty. These will be designated in this report as Al-Si, No. 1, No. 2 and No. 3, respectively. The compositions of these four alloys are summarized in Table XI.

The composite core sand and green sand mold (Fig. 3) was used for the test castings. In this composite mold, the gate, the end near the gate, and two-thirds of one side of the mold were made of core sand, the balance being rammed from green sand. With such a mold one face of the casting will illustrate the behavior of the metal in contact with core sand and the opposite face, the

Table XII
Tensile Properties of Steel Castings Deoxidized with Special Deoxidizers Consisting of Al-Si-Fe Alloys

All castings were made in combination molds having green-sand and core-sand faces. Heat 6702, composition, C, 0.20 per cent; Mn 0.69 per cent; Si 0.38 per cent.

Specimen Number[1]	Additions[2]	Silicon[3] Per Cent	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
1B	none		41,000	71,500	25.0	31.5
1T			44,500	71,500	28.5	41.3
2B		.38	43,500	71,500	29.0	37.5
2T			43,500	71,500	25.0	32.5
Average			42,800	71,500	27.0	35.5
3B	aluminum		45,000	73,500	18.5	22.5
3T			43,500	72,000	17.0	21.0
4B		.38	45,000	72,000	21.0	21.5
4T			44,000	72,000	18.0	19.5
Average			44,200	72,300	18.5	21.0
5B	Alsifer		44,500	73,000	14.0	27.5
5T			44,000	72,000	17.0	19.5
6B		.49	43,000	72,000	20.0	23.5
6T			43,500	71,000	17.5	21.5
Average			43,700	72,000	19.5	23.0
7B	Al-Si alloy		42,000	72,000	23.5	28.0
7T	No. 1		43,000	71,000	20.5	21.0
8B		.46	42,000	72,000	22.5	25.0
8T			42,000	70,000	17.5	22.5
Average			42,000	71,200	21.0	24.0
9B	Al-Si alloy		40,500	71,000	22.5	28.0
9T	No. 2		40,000	70,000	18.5	24.0
10B		.48	44,500	70,500	20.5	27.0
10T			44,000	70,000	18.5	24.0
Average			42,000	70,400	20.0	25.5
11B	Al-Si alloy		41,000	72,500	20.5	23.5
11T	No. 3		40,500	71,000	22.5	27.5
12B		.68	44,500	71,000	22.5	26.5
12T			44,500	72,000	16.0	21.5
Average			42,600	71,600	20.5	25.0

[1]B = Bottom, T = Top specimen as cut from castings.
[2]All additions made in the 70-pound hand ladle; the amount being equivalent to 1 pound aluminum per ton of steel.
[3]Silicon analysis after Al-Si alloy additions.

behavior of the metal in contact with green sand. At the same time the reduced section from which the tensile test bars are cut gives the properties of green-sand castings. Fig. 13 shows the appearance of one of the test castings from both the green sand and core sand faces after chipping the test casting at the fillets. (Fig. 3.)

Some pin holes were found after chipping the casting at the fillets but the only castings which showed an appreciable degree of porosity in the fillets were those made without the addition of aluminum. Porosity was found on the green sand side. The same metal in contact with the core sand appeared to be perfectly sound as is shown by the two photographs in Fig 13.

In order to examine further the condition of the metal, a 1-inch section from the end opposite the gate was cut from each test

Table XIII
Comparative Tensile Properties of Steel Castings Deoxidized by Aluminum and by an Al-Si-Fe Alloy Added in Different Manners

Castings were made in combination green-sand core-sand molds. Heat 6976; composition, C 0.20 per cent; Mn 0.73; Si 0.35 per cent, heat treated by normalizing, 1650 degrees Fahr. (900 degrees Cent.)—2 hrs.

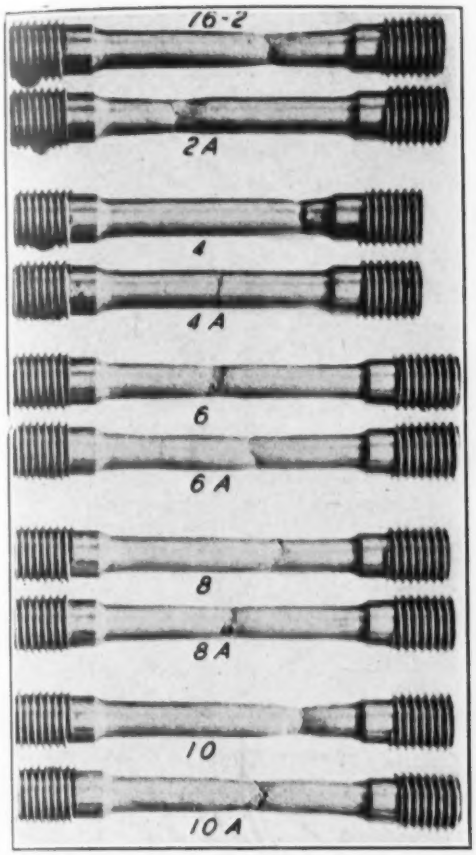
Specimen Number	Addition[1]	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area	Remarks
76– 2	none	42,000	76,000	27.0	41.5	rubber bonded sand used on
2a	none	42,000	77,000	27.5	40.0	fillet; sound casting.
4	none	.....	.....	....	....	porous
4a	none	.....	.....	....	....	porous
6	Aluminum[1]	40,000	75,500	28.5	34.5	sound
6a	Aluminum[1]	36,000	75,000	28.5	38.5	sound
8	Al-Si-Fe[2] alloy in 70-lb. ladle	38,000	75,000	28.5	39.0	sound
8a	Al-Si-Fe[2] alloy in 70-lb. ladle	37,000	74,000	21.0	33.0	sound
10	Al-Si-Fe[2] alloy in 2½–ton ladle	37,000	71,500	26.0	48.5	sound
10a	Al-Si-Fe[2] alloy in 2½–ton ladle	36,500	71,500	26.0	43.0	sound

[1]Equivalent to 1 lb. aluminum per ton of steel.
[2]Composition of Al-Si-Fe alloy, as given by manufacturers—
Al 45 to 50 per cent
Si 30 to 35 per cent
Fe balance

casting, the cut being continued through the casting and the head. All of the test castings were found to be sound except the one that did not contain aluminum.

The results of the tensile tests of these castings are summarized in Table XII. While the particular heat selected for these experiments showed relatively low values of reduction of area when cast without the addition of aluminum, the results clearly indicate a reduction in the ductility upon adding aluminum either in the metallic form or as part of any of the four aluminum-silicon-iron alloys used. In general, the tests showed that the addition of each of the four aluminum-silicon-iron alloys in amounts equivalent to 1 pound of aluminum per ton of steel produced sound castings but lowered the ductility to about the same degree as did the addition of the aluminum alone.

Some further information on the results attainable by the use of the aluminum-silicon-iron alloy as a deoxidizer is given in Table XIII and Fig. 14. In this case the alloy was added in the small working ladle in one case and in the 2½ ton ladle in another. The results as shown by both the data in Table XIII and the appearance of the bars after testing (Fig. 14), were distinctly more favorable to the use of this deoxidizer than were the previous ones.



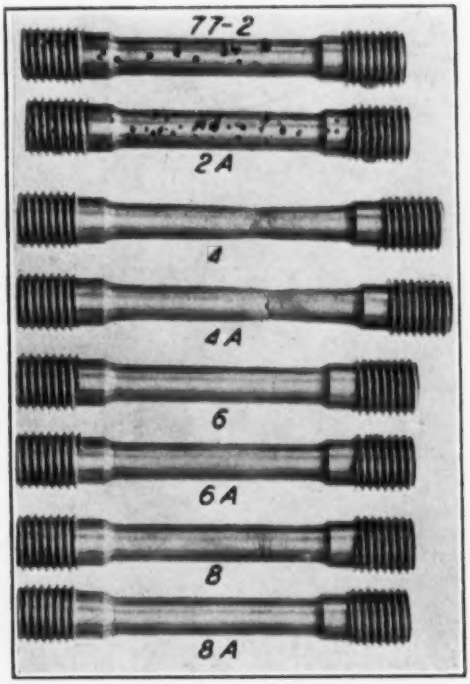


Fig. 14 (Left)—Tensile Specimens, after Testing, from Steel Test Castings "Deoxidized" by the Al-Si-Fe Alloy (Table XV). ×½. Reduced about ⅛ in Printing. Specimens 76-2, 2A, 4 and 4A, not Deoxidized; 6 and 6A, Deoxidized with 1 lb. Al per Ton of Steel; 8, 8A, 10 and 10A, Deoxidized with Al-Si-Fe Alloy Equivalent to 1 lb. Al per Ton of Steel.

Fig. 15 (Right)—Tensile Specimens, after Testing, from Steel Test Castings "Deoxidized" by the use of Aluminum and an Alloy of Manganese, Silicon and Boron. ×½. Reduced about ⅛ in Printing. Specimens 77-2, and 2A, no Deoxidizer; 77-4 and 4A, 1 lb. Al per Ton of Steel; for the Remainder the Mn-Si-B Alloy was Used (Table XVI).

Another point of interest is improvement which resulted from the use of rubber-bonded sand instead of green sand in making the mold. The tendency of the steel containing no aluminum toward porosity was very decidedly reduced by its use.

A number of other alloys and other additions which appeared to offer some possible promise as a deoxidizer were tried. Among these were an alloy of manganese-silicon-boron, alloys of aluminum, silicon and boron, of several different compositions, metallic beryllium, an iron-aluminum alloy, and ferrotitanium. In addition a number of rather unusual additions were used consisting of lead, brass (80 per cent copper, 20 per cent zinc), copper, zinc, and

Table XIV

Tensile Properties of Test Steel Castings Deoxidized with Aluminum or a Mn-Si-B Alloy

All castings were made in the combination green-sand core-sand mold. Heat 6977; Composition, C 0.24 per cent; Mn 0.73 per cent; Si 0.35 per cent, heat treated by normalizing, 1650 degrees Fahr. (900 degrees Cent.)—2 hours

Specimen Number	Additions	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area	Remarks
77–2	none	.....	.....	....	....	porous
2a	none	.....	.....	....	....	porous
4	1 lb. Al per ton steel in 70-lb ladle	38,000	66,000	24.0	33.0	sound
4a	1 lb. Al per ton steel in 70-lb. ladle	40,000	67,000	27.0	42.0	sound
6	4 oz. of alloy F[1]	36,000	61,500	7.0	6.0	sound
6a	70-lb. ladle[2]	broke in fillet		....	....	sound
8	20 lbs. of alloy F made in 2½-ton ladle[3]	31,500	60,000	....	....	sound
8a		28,000	46,000	....	....	sound

[1]Composition of Alloy F—Mn 45%, Si 25%, B 24%.

[2]Equivalent percentage of	Mn	Si	B
approx.	.16	.09	.08
[3]Equivalent percentage of	.18	.10	.096

fused soda ash. The results of the tensile tests of the resulting castings are given in Tables XIV to XVIII inclusive and in Figs. 15, 16 and 17. In a number of cases, the appearance of the tested tensile specimen was much more indicative of the character and condition of the metal than the numerical results.

In Table XIV and Fig. 15 are shown the results obtained by using an alloy of manganese, silicon and boron as a deoxidizer.

Results showing the effect of additions of an alloy of aluminum, boron and silicon as a "deoxidizer" are given in Tables XV and Fig. 16. Alloys of three different compositions were used and for comparison, the results obtained by adding aluminum in the ordinary manner and also the results of one series in which metallic beryllium was used as the deoxidizer. It will be noted that the bars treated with the aluminum-boron-silicon alloy, though sound, were exceedingly low in their tensile properties and that those of the beryllium treated bars were only on a par with the aluminum treated ones.

The effect upon the tensile properties of steel castings produced by a number of special additions, including boron, a boron-manganese-silicon alloy, a mixture of ferrosilicon and ferromanganese, an aluminum-iron alloy and carbon-free ferrotitanium is shown in Table XVI and Fig 17. Of the various additions, the

Table XV

Tensile Properties of Steel Test Castings Deoxidized in Various Ways, with an Alloy of Aluminum, Boron and Silicon, with Aluminum and with Beryllium

Castings were made in the combination green-sand and core-sand mold.
Heat 6981; C 0.24 per cent, Mn 0.72 per cent, Si 0.30 per cent; normalized at 1650 degrees Fahr. (900 degrees Cent.) 2 hours.

Specimen Number	Additions	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area	Remarks
81– 2	none	.....	.....	....	....	porous
2a	none	.....	.....	....	....	porous
4	1 lb. Al per ton of steel	35,500	71,500	26.0	30.0	sound
4a	(.05% Al)	36,000	71,000	24.0	26.0	sound
6	½ lb. Al per ton of steel	40,000	73,500	28.0	44.5	sound
6a	(.025% Al)	39,500	73,500	28.0	44.0	sound
8	½ lb. of Be per ton of	38,000	71,000	27.5	40.0	sound
8a	steel (.025% Be)	37,500	69,500	26.5	36.0	sound
10	500 grams alloy A[1]	30,000	55,000	broke in threads		sound
10a		35,000	59,000	broke in fillet		crystalline structure
12	125 grams alloy[1] A	30,000	52,000	broke in fillet		crystalline structure
12a		33,000	55,000	broke in fillet		crystalline structure
14	110 grams alloy[1] B	45,000	50,500	broke in threads		crystalline structure
14a		48,000	55,500	low ductility		crystalline structure
16	27 grams alloy[1] B	32,000	67,000	17.5	16.0	crystalline structure
16a		32,000	63,000	12.0	7.5	crystalline structure
18	40 grams alloy[1] C	40,000	49,000	broke in fillet		crystalline structure
18a		35,000	45,500	low ductility		

[1]Chemical composition of alloys A—Boron 20.18 Aluminum 3.06 Silicon 4.90
B— 16.77 14.87 1.26
C— 10.50 13.50 12.5

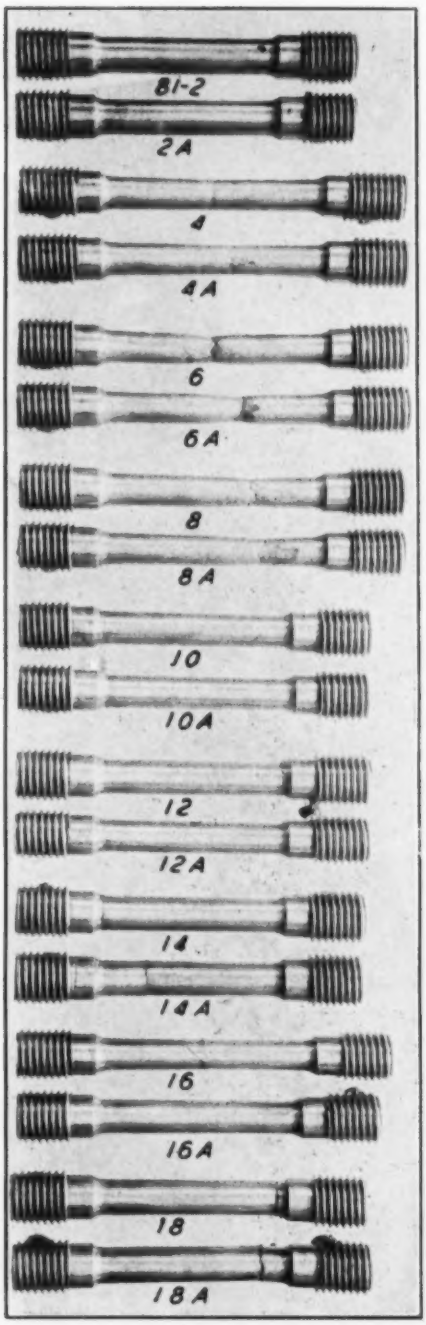
Additions were made in 70 pound ladle.
Approximate percentage additions of

	B	Al	Si
Specimen 10	.31	.05	.08
Specimen 12	.08	.015	.02
Specimen 14	.06	.05	.04
Specimen 16	.014	.012	.013
Specimen 18	.013	.016	.015

mixture of ferrosilicon and ferromanganese was the only one which appeared to be promising.

Additional information on the effect of titanium as a deoxidizer for steel castings when added in the form of ferro carbon titanium is given in Table XVII. It will be noted that the ductility of the treated steel castings was much better in this case than in the tests included in Table XVI, in which carbon-free ferrotitanium was added to the small ladle instead of the large ladle, although the amount of titanium added did not differ greatly in the two cases. The test castings, however, were sound.

A few additional tests on deoxidizers were made with a ferro



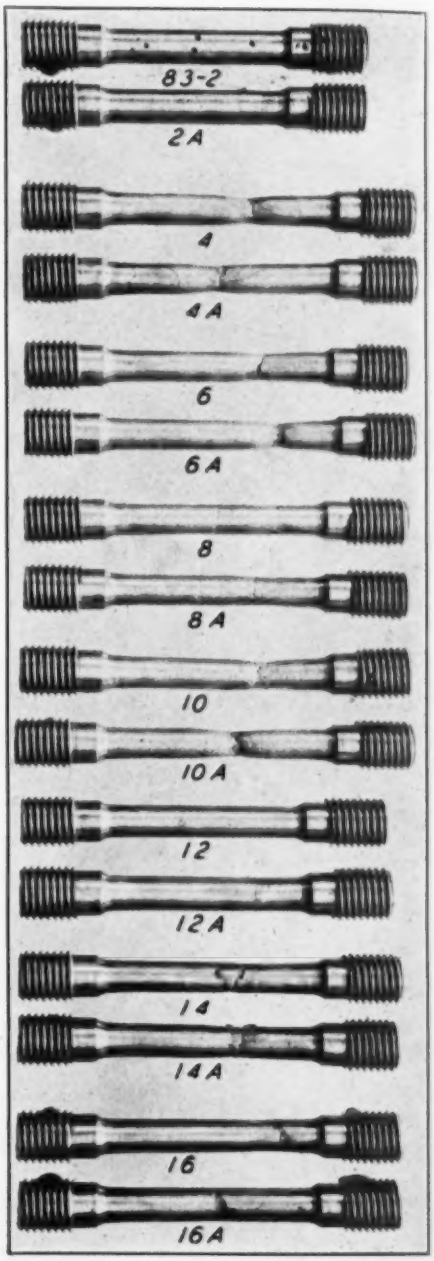


Fig. 16 (Left)—Tensile Specimens, after Testing, of Steel Test Castings Deoxidized in Various Ways. ×½. Reduced about ⅛ in Printing. Specimens 81-2 and 2A, no Addition; 81-4 and 4A, 1 lb. Al per Ton of Steel; 81-6 and 6A, ½ lb. Al per Ton of Steel; 81-8 and 8A, ½ lb. of Be per Ton of Steel; the Remainder Were Treated with Alloys of Al-B-Si of Various Compositions (Table XVII).

Fig. 17 (Right)—Tensile Specimens, after Testing, of Steel Test Castings Treated with Special Deoxidizers. Specimens 83-2 and 2A, no Addition; 83-4 and 4A, 1 lb. Al per Ton of Steel; 83-6 and 6A, ½ lb. Al per Ton of Steel; 83-8 and 8A, Boron; 83-10 and 10A, Mixture of Ferrosilicon and Ferromanganese; 83-12 and 12A, B-Mn-Si Alloy; 83-14 and 14A, Fe-Al; 83-16 and 16-A, Carbon-Free Fe-Ti.

Table XVI

Effect on the Tensile Properties of Steel Castings of a Number of Special Deoxidizers Including Aluminum, Boron, Boron-Silicon-Manganese Alloy, Aluminum-Iron Alloy, a Mixture of Ferrosilicon and Ferromanganese, and Carbon Free Ferrotitanium

All castings were made in the combination green-sand and core-sand molds.
Heat 6983; C 0.23 per cent, Mn 0.66 per cent, Si 0.31 per cent; heat treated by normalizing at 1650 degrees Fahr. (900 degrees Cent.) 2 hours.

Specimen Number	Additions[1]	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area	Remarks
83–2	none	.....	.....	....	....	porous
2a	none	.....	.....	....	....	porous
4	1 lb. Al per ton of steel	35,000	70,500	30.0	46.0	sound
4a	(.05% Al)	36,000	69,000	30.0	46.0	sound
6	½ lb. Al per ton of steel	39,000	71,000	31.0	51.5	sound
6a	(.025% Al)	36,500	70,500	28.5	40.0	sound
8	40 grams alloy D[2]	33,500	71,600	20.0	18.0	sound
8a	(.007% B)	34,500	70,000	20.0	22.5	sound
10	mixture of ferrosilicon and	30,000	70,000	26.0	40.0	sound
10a	ferromanganese[3]	38,500	72,000	34.0	51.0	sound
12	70 grams alloy F[2]	38,000	62,500	6.0	6.0	sound
12a	(.05% B, .06% Si, .10% Mn)	39,000	67,000	10.0	10.0	sound
14	40 grams FeAl[4]	35,500	67,000	20.0	22.0	sound
14a	(.06% Al)	36,000	67,000	20.0	28.0	sound
16	30 grams C free FeTi[2]	38,000	68,000	18.5	18.0	sound
16a	(.025% Ti)	39,500	68,000	19.0	21.5	sound

[1]Additions were made in the 70-pound ladle.
[2]Chemical composition of alloys D— Boron 6.0 Si .... Mn ....
F— 24.0 25.0 45.0
FeTi—approximately 28% Ti
[3]Addition equivalent to .05% Si and .10% Mn in the steel.
[4]FeAl made by melting 10 pounds of aluminum and placing pot on the scales, then adding 10 pounds of molten steel, allowed to cool without stirring.

Table XVII

Tensile Properties of Steel Test Castings Deoxidized with Ferro-Carbon-Titanium

Three pounds of ferro carbon titanium, containing approximately 20 per cent Ti, per ton of steel were added in the large "teapot" ladle. No aluminum was used.
Castings were normalized 2 hours at 1650 degrees Fahr. (900 degrees Cent.)

Heat No.	Per Cent C	Per Cent Mn	Per Cent Si	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
3990	0.26	0.70	0.48	43,500	77,000	31.0	48.0
3991	.22	.70	.45	42,000	79,000	27.0	44.0
3992	.28	.89	.60	44,000	88,000	27.5	48.0
3994	.16	.74	.39	43,500	77,000	31.0	54.6
3996	.20	.69	.40	42,500	77,500	28.0	44.5
3997	.20	.75	.44	48,500	83,000	26.0	46.0

alloy of aluminum-silicon-titanium. This alloy contained aluminum 8 per cent, silicon 20 per cent, titanium 5 per cent. Additions of this alloy did not seriously impair the ductility of the resulting castings but the castings were porous. Experiments were also made with a silicon-zirconium alloy. This was added to the large ladle

Table XVIII
Tensile Properties of Steel Test Castings Treated with Various Unusual Additions

Heat 5589; C 0.21 per cent, Mn 0.59 per cent, Si 0.35 per cent

Specimen Number	Deoxidation[1] Addition	Amount Lb. Per Ton of Steel	Tensile Properties: Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area	Remarks
1	none	...	40,000	71,000	17.0	35.5	pin holes
2			40,000	67,000	12.5	30.0	
3	Al	1	39,000	67,500	25.0	32.0	sound
4			41,000	68,000	28.0	33.0	
5	Pb	3.7	43,500	69,500	15.0	24.0	pin holes
6			43,500	67,000	13.0	23.0	
7	80:20 brass	3.7	45,000	72,000	18.0	22.5	pin holes
8			43,000	67,000	15.0	29.0	
9	Cu	3.7	43,500	72,000	20.0	29.0	pin holes
10			41,500	67,500	18.5	24.0	
11	Zn	1	42,000	70,500	34.0	52.0	sound
12			38,500	71,000	33.0	55.2	
13	"purite"[2]	7.5	39,500	70,000	18.0	31.0	pin holes
14			43,000	72,000	37.5	52.0	

[1]All additions were made in the 70-pound ladle except the "purite" which was added in the 300-pound ladle.

[2]Fused sodium carbonate. This is ordinarily added to remove sulphur rather than as a deoxidizer.

Table XIX
Effect of Different Mold Surface Treatments on the Tensile Properties of Steel Test Castings

Heat 37-56 of Company "B"; C 0.23, Mn 0.79, Si 0.44, P 0.026, S 0.038 per cent; castings were normalized at 1650 degrees Fahr. (900 degrees Cent.) 2 hours and reheated 2 hours at 1250 degrees Fahr. All castings were made in combination green-sand core-sand molds, but specimens were from the green-sand portion of the mold.

Specimen Number	Addition to Steel and Treatment of Molds	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area	Remarks
2	none	.....	.....	....	....	porous
2a	none	.....	.....	....	....	porous
4	1 lb. Al per ton of steel equivalent	43,500	70,500	27.5	37.5	sound
4a	to .05% Al	43,000	70,000	27.5	36.5	sound
6	painted with a rubber preparation[1]	36,000	72,000	34.0	56.5	sound
6a		37,500	72,500	35.0	55.5	sound
8	painted with a rubber preparation[1]	.....	.....	....	....	porous
8a	and silica flour	.....	.....	....	....	porous
10	painted with a rubber preparation[1]	.....	.....	....	....	porous
10a	and graphite	.....	.....	....	....	porous
12	painted with Mg powder and gasoline	.....	.....	....	....	porous
12a	and skin dried	38,000	72,500	35.5	55.5	sound
14	dusted with dry Mg powder	.....	.....	....	....	porous
14a	dusted with dry Mg powder	42,000	68,500	35.0	60.5	sound
16	dusted with Al powder	38,000	68,000	35.0	62.0	sound
16a	dusted with Al powder	38,000	68,000	36.0	55.5	pin holes

[1]An 8 per cent gasoline solution of "Vulcalock" cement—a thermoprene compound.

(2½ tons). The results obtained with an addition of zirconium of 0.10 per cent per ton of steel were favorable from the standpoint of porosity but the ductility of the steels was decreased about the same as with aluminum addition of 1 pound per ton.

Table XXA

Effect of Sand Control and Heat Treatment on the Tensile Properties of Cast Steel Made in Green Sand Molds—12 Heats of 2½ Tons Each

Foundry "B" deoxidized in large ladle with aluminum equivalent of 0.6 pounds per ton.
Foundry "A" deoxidized in shank ladle with aluminum equivalent of 1 pound per ton.

		Composition Per Cent			Normalized at 1650°F.—2 hours				Normalized at 1650°F.—2 hours and Drawn at 1250°F.—2 hours			
Heat No.	Specimen	C	Mn	Si	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area	Yield Point Lbs. Per Sq. In.	Tensile Strength Lbs. Per Sq. In.	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
Foundry "B"												
188	1T[1]	.18	.63	.36	37,500	69,000	31.0	55.0	36,500	69,000	35.0	52.0
	1B[2]				38,000	68,000	31.0	53.5	39,000	69,000	34.5	58.5
189		.19	.71	.35[3]								
190	3T	.20	.79	.33	37,500	70,500	37.0	56.0	39,000	68,500	32.5	58.0
	3B				43,000	75,000	31.0	56.0	39,000	69,500	32.5	58.5
191	4T	.22	1.06	.37	42,000	85,000	22.5	33.0	45,000	84,000	27.5	48.0[4]
	4B				41,500	84,500	27.5	57.5	46,500	84,000	31.0	59.0
192	5T	.20	.75	.35	42,500	71,500	32.5	53.0	41,500	71,500	32.5	60.0
	5B				45,500	71,000	32.5	59.0	41,500	71,500	29.0	48.0
193	6T	.25	.97	.38	44,000	82,000	20.0	31.5	44,000	71,000	29.0	49.5
	6B				45,000	82,000	25.0	39.5	43,000	80,500	25.0	35.5
Foundry "A"												
8862	7T	.21	.68	.45	44,000	73,000	18.0	21.5	41,500	73,000	27.5	32.5
	7B				47,000	74,000	22.5	30.5	47,000	74,000	27.5	37.5
8863	8T	.19	.62	.34	36,000	68,000	29.0	40.5	36,500	68,500	31.0	46.5
	8B				47,000	69,500	30.0	39.5	38,000	68,500	30.5	46.5
8864	9T	.20	.60	.30	46,000	69,500	22.0	29.0	41,000	68,000	27.0	37.0
	9B				35,000	69,000	30.0	44.0	42,000	68,500	29.0	40.0
8865	10T	.18	.65	.36	45,000	71,500	28.0	35.0	47,000	71,000	28.0	42.0
	10B				46,000	71,500	29.0	42.0	45,000	71,000	28.0	42.0
8866	11T	.20	.67	.31	44,000	69,000	24.0	37.5	43,500	69,000	27.5	35.0
	11B				42,000	69,000	28.0	42.0	39,500	64,000	27.5	38.5
8867	12T	.21	.74	.33	43,500	72,000	27.5	32.5	44,000	70,500	23.0	30.0[4]
	12B				44,000	72,000	30.0	42.0	42,000	72,000	28.0	34.5[4]

[1]T represents specimen from top of test casting.
[2]B represents specimen from bottom of test casting.
[3]Heat 189 rejected—porous.
[4]Defective specimen.

Table XXB

Characteristics of Green Sand Facings Used for Above Heats of Test Castings

Foundry	Moisture Per Cent	Permeability Cu. Cm. Air Per Min. Per Cu. Cm. of Sand	Compressive Strength Lbs. Per Sq. In.	Sieve Analysis Per Cent of Sand Retained on Mesh (U. S. Standard) 6	12	20	40	70	100	140	200	270	Pan	Grain Clay[1]
A	2.0	204	6.9	..	0.40	1.65	9.65	54.05	20.40	3.40	1.15	0.35	1.30	92.35 6.7
B	2.1	291	5.6	..	0.10	2.50	32.25	44.30	10.10	2.10	0.80	0.30	0.20	92.70 6.3

[1]Determined by washing test.

Table XVIII shows the effect of some miscellaneous additions on the tensile properties of test castings. Most of these would not ordinarily be classed as deoxidizers. These results show that all of the treated bars contained "pin holes" except those treated with aluminum.

3. Surface treatment of green sand molds

The assumption underlying all of the previous work covered by this report is that the moisture of the green sand mold is primarily responsible for the difficulties which are experienced. As stated at the outset, by the use of dry sand molds in steel casting work, such difficulties are reduced to a minimum. A few experiments were carried out for the purpose of showing whether or not, by suitable mold preparations, the difficulties accompanying the use of green sand molds could be overcome by counteracting the effect of the moisture in such molds. The mold preparations used consisted of various surface treatments. The results are summarized in Table XIX.

4. Effect of green sand control

From the beginning of this investigation, it was believed that variables in green sand practice, especially moisture, were largely responsible for the difficulties encountered from "pin-holes" in most steel foundries. It was thought, however, that sand control would be harder to maintain to a high degree than would some suitable deoxidizer which might insure soundness without impairing ductility. The early work on deoxidizers, however, was not especially promising so the effect of sand control was considered.

Molds were made from sand having the characteristics as shown in Table XXB. Instead of using a facing sand containing the usual amount of clay binder (bentonite), some of this binder was replaced by cereal bonds (corn flour and wheat flour) which varied between 0.2 and 0.6 per cent by volume, while the bentonite varied between 0.4 and 0.8 per cent. Molasses water, consisting of 1 part molasses to 3 parts of water, was used for tempering. It is of interest to note that the sand characteristics used for these experiments are quite different from those used for the early work (Table II). In this latter work, however, the sand samples were taken from extra molds, air dried in the same manner as the molds for the test castings and not from molders' bins.

The tensile properties of twelve heats (Table XXA) showed that little difficulty was encountered from porosity or low ductility when the moisture and permeability of the sand was closely controlled and when the carbon content of the heat varied between 0.18 and 0.25 per cent. The six heats from Foundry B had higher

ductility after the heat treatments than those from Foundry A. Aluminum was added in both cases but less was added to the heats from Foundry B; also this foundry made the additions to the large ladle by the submersion method previously described. This allowed more efficient deoxidation and also more time for the deoxidation products to escape from the metal, which may have been the reason for the increased properties.

V. Summary

1. The tensile properties frequently specified for acid electric steel castings, as minimum requirements, are an ultimate tensile strength of 60,000 pounds per square inch, an elongation in a 2-inch gage length of 24 per cent, and 35 per cent reduction of area. When the test castings are made in dry sand molds no difficulty is encountered in meeting these requirements, but when green sand molds are used, the castings often lack soundness. This difficulty is usually overcome by the addition of a deoxidizer, usually aluminum, shortly before the metal is poured, but the test castings in this case not infrequently fail to meet the specification requirements with respect to ductility. A study of this problem was undertaken by the Bureau at the request of and with the co-operation of the Steel Castings Development Bureau on the research association plan. The tensile properties and soundness of test castings of acid electric steel made in several steel foundries under a variety of conditions have been studied at considerable length.

2. A rather detailed preliminary survey of the field was made by the study of 100 consecutive heats made under observed conditions in one foundry and 32 made in one other to illustrate the extent of the problem. The tendency toward low ductility is related to the carbon content of the steel. This preliminary survey indicated, in general, that if the carbon content is maintained between 0.10 and 0.17 per cent, the manganese between 0.60 and 0.70 per cent and the silicon content between 0.30 and 0.40 per cent, the addition of about 1 pound of aluminum per ton of steel to the working ladle shortly before pouring will not lower the ductility of the normalized castings below the usual specified limits.

3. A study of the different variables in ordinary acid electric steel foundry practice indicated that moderate changes in these variables have very little effect with respect to the matter of low

ductility of the steel. Variations in the size and character of the scrap used in the charge have no appreciable effect. Alloying additions of nickel and of vanadium in moderate amounts do not appear to improve the ductility. Changes in heat treatment, aside from rather pronounced changes involving quenching and tempering which are not practicable in ordinary foundry practice, appeared to have no appreciable effect upon the low ductility of castings. There was some indication, however, that the use of a higher normalizing temperature, for example, 2000 degrees Fahr. (1100 degrees Cent.) instead of 1650 degrees Fahr. (900 degrees Cent.) is beneficial. The use of a rather high casting temperature appears to be associated with higher ductility.

4. Of a rather large number of additions used as possible deoxidizers, none were superior to aluminum in producing soundness. Alloy deoxidizers containing aluminum produce essentially the same effect as the equivalent amount of aluminum. Aluminum and aluminum alloys do not reduce the ductility any more than the other types of effective deoxidizers used such as ferroboron, silicon, zirconium and ferrotitanium. A considerable number of the additions used as possible deoxidizers were found to have a very marked effect in reducing the ductility of the steel.

5. The relation between the amount of aluminum added to the steel and the resulting effect upon the ductility is not a linear relationship. The amount of aluminum necessary to insure soundness in green sand molds without seriously impairing the ductility may vary for different steel according to the melting practice, the mold condition, and the state of oxidation of the steel when tapped. It is believed, however, that a sufficient amount of "deoxidizer" should be in the steel at the time of pouring to deoxidize the steel from the gases evolved by the green sand. With normal practice the equivalent of about ½ pound of aluminum per ton of steel added in the form of metallic aluminum or as an alloy of aluminum will usually give sound castings which after normalizing will show ductility somewhat better than the specified minimum. If the aluminum is added during the tapping, the resulting castings may be somewhat porous. The addition of at least part of the aluminum immediately before pouring is recommended. Although there is no definite proof, the inference from this work that inclusions of Al_2O_3 are largely responsible for the low ductility re-

sulting when aluminum is added to the steel shortly before casting would seem to be justified.

6. The results of a few experiments with mold preparations in the form of surface treatments for counteracting the influence of the moisture in green sand molds indicated that this method has merit but the results were not especially promising.

7. The situation as regards porosity can generally be improved and the difficulties often overcome by controlling the mold conditions, as regards moisture and permeability. By this method the minimum of aluminum or other deoxidizers are required to compensate for the gases evolved by the mold. If the carbon content is about 0.20 per cent it will then be easier to meet the ductility specifications.

ACKNOWLEDGMENTS

The authors wish to express their appreciation and thanks to the Steel Castings Development Bureau and to George Batty, director of Research of that Bureau, for their interest throughout this investigation. Previous to the organization of the Steel Castings Development Bureau, the most hearty co-operation and support was given by the following persons: Clarence Tolan, Jr., and C. S. Roberts of Dodge Steel Co., H. C. H. Cunningham and C. R. H. Cunningham of Crucible Steel Casting Co. We also wish to express our sincere appreciation to H. S. Rawdon, chief of the Division of Metallurgy, and to Dr. H. W. Gillett, former chief of the Division of Metallurgy of the Bureau of Standards for their helpful suggestions, to H. J. French, formerly of the Bureau of Standards for directing the early part of the investigation, to the metallurgical staff as a whole, but more especially to H. K. Herschman, S. J. Rosenberg, L. Jordan and C. M. Saeger, Jr. We also wish to extend our appreciation to Dr. C. H. Herty, Jr., and J. M. Gaines, Jr., of the Bureau of Mines for co-operation and helpful suggestions.

DISCUSSION

Written Discussion: By L. E. Gilmore, Crane Co., Chicago.

The comparison of the effect on steel of green sand and dry sand in the same mold is a happy idea and a convincing exhibit.

While the experiments made in this investigation were on acid electric steel, the conclusions probably apply to a large extent to basic electric practice also.

In basic operation we have found that the use of various scrap charges,

such as heavy or small sized melting stock or of sand blasted or milled scrap and the like, had no particular effect on physical properties of finished steel. If alumina or other inclusions are granted to be the cause for reduced ductility, the explanation why the nature of the initial charge has little or no effect on physical properties is evident from the following observations made on the presence of sonims (solid nonmetallic inclusions) at different stages during a steel heat.

Immediately after the boil, samples cast from the bath were practically free from sonims. A moderate boil almost completely freed the bath from sonims no matter what the nature of the original charge.

After the addition of silicon and manganese shortly before tapping, samples cast showed considerable increase in sonims. The addition of aluminum in the ladle also tended to add to the inclusions.

From this it appears that not only aluminum but also other additions may have some part in producing sonims with consequent effect on ductility in the finished steel.

Written Discussion: By W. C. Hamilton, research director, American Steel Foundries, Chicago.

One who studies this paper must realize the vast amount of work which is represented, and the authors are to be congratulated for having submitted such very interesting and instructive information.

The sponsors of this work have encountered porosity in green sand castings poured from acid electric steel. In order to overcome this porosity, aluminum or other deoxidizers have been found a necessity. Aluminum has been most generally used, and it promotes solidity but greatly impairs the ductility of the steel.

The factors which were included in this investigation are listed below:

1. Furnace charges
2. Furnace operations
3. Methods of making additions
4. Deoxidizers
5. Use of alloys
6. Casting temperatures
7. Type of molds
8. Composition
9. Heat treatments

The conclusions reached are summarized as follows:

First—There were no solutions offered to the problems from a study of furnace charges, furnace operations, methods of making additions, or the use of alloys.

Second—It was found that if the carbon content of the steel is 0.20 per cent or under the ductility specifications will be more easily met.

Third—The results obtained indicate that the influence of moisture and permeability in green sand molds is very great, and that when these conditions are correct the amount of deoxidizer can be reduced in order to obtain better ductility.

Fourth—It has been shown that no other deoxidizer was superior to aluminum in producing solidity.

Fifth—It is intimated that a high casting temperature is associated with higher physical properties, including tensile strength and ductility.

Sixth—It has also been intimated that a normalizing treatment at approximately 2000 degrees Fahr. is beneficial.

The results reported do not conclusively show that a high casting temperature and a high temperature for normalizing are really of benefit, and additional work would seem necessary before these practices should be accepted as being of value.

The influence of moisture and permeability on the porosity of castings poured in green sand molds is known to be a very decided one, and it is not our intention to minimize these effects. Also, a low carbon content in steel promotes greater ductility.

High casting temperatures are discussed on page twenty-one. It is shown that the tensile strength of steel increases with the casting temperature. A great advantage of electric steel is the fact that it can be poured into thin sections, and this is due, at least partially, to a high casting temperature. The higher tensile strength obtained from electric steel is probably due, at least in part, to the higher casting temperatures. It has been our experience that the same tensile strength can be obtained from acid electric steel and acid open-hearth steel with a carbon content of 0.20 per cent in the former case as compared to a carbon content of 0.26 per cent in the latter case.

On the seventeenth page are given tensile properties of cast steels with varying heat treatments, and there are a number of inconsistent results tabulated. Duplicate bars show a decided difference in several cases, but more striking are the results from heats 4924 and 4981. Bars from these heats were normalized at 1650 degrees Fahr. and the tensile strength in one case is higher from heat 4924 containing 0.15 per cent carbon than from heat 4981 containing 0.26 per cent carbon.

On the twelfth page there is given an example of carbon differences found in steel castings, and it is intimated that this is an occurrence common to steel castings only. Our experience does not substantiate this opinion in connection with either open-hearth or electric steel when made and tapped under proper conditions.

The title of the paper covers carbon steel castings, but no mention is made of any other than acid electric steel in the contents. Our experience has been largely limited to open-hearth steels and the difficulties brought out in this paper had not been encountered by us until we began the manufacture of acid electric steel castings.

At first no aluminum additions were made and the castings were porous, whether poured in green sand or dry sand molds. The use of aluminum in various forms was investigated and the preliminary results showed very poor ductility in the steel. A comprehensive study was made and it became possible to predict the ductility which would be obtained from the composition and type of inclusions present. From this study was

developed an acid electric furnace practice whereby solid castings were obtained with the addition of ten ounces of aluminum to the ton of steel, or with an equivalent amount of aluminum added as an alloy, such as Alsifer.

A typical analysis with physical results after a normalizing at 1650 degrees Fahr. is as follows:

Analysis:

C	Mn	Si	P	S
0.23	0.69	0.40	0.028	0.034

Physical results:

Elongation	29.0%
Reduction of area	50.2%
Yield point	43,220 lbs. per sq. in.
Tensile strength	74,760 lbs. per sq. in.

As already noted, our problem included porosity in castings poured in dry sand molds when acid electric steel was produced, and this was overcome by changes in furnace practices rather than by changes in foundry practices.

The main change, before it was possible to obtain sound castings and satisfactory ductility, was in our idea of maintaining a reducing condition in an acid electric furnace. Oxidation in open-hearth practice, as usually followed, is a necessity, and it was found that many of the practices followed in open-hearth furnaces could be used to great advantage in the acid electric furnace. The important points in this electric furnace practice as followed by us are as follows:

The bath should contain from 0.30 to 0.40 per cent carbon when melted.

During melting, and when pools of metal are forming, there should be added five shovels of used foundry sand in order to have formed a protective slag at an early period.

A five-ton heat is melted in approximately one hour.

One-half hour after charging, additions of iron ore are made. This ore is added in fifty pound lots at intervals of five or ten minutes until a total addition of two hundred pounds is made.

In about thirty minutes after the last addition of iron ore the boil has ceased and tests for analysis and solidity are poured; then if the bath is in condition the additions are made.

First is added the 80 per cent ferromanganese, and after five minutes the 50 per cent ferrosilicon. In another ten minutes the heat is tapped.

To the stream of metal entering the ladle is added fifteen pounds of Alsifer or three pounds of aluminum, together with twelve pounds of 50 per cent ferrosilicon.

It should be noted that iron ore is the essential addition during the melting, or immediately following it, rather than coke or pig iron, and that no lime is used in our practice. This addition of iron ore results in a better action on the bath and a more complete elimination of the residual silicon.

With the practice outlined above it has been possible to get very consistent physical results such as before quoted, even with an addition of aluminum equivalent to ten ounces per ton of charge. Attention is also called to the fact that it has been found possible to obtain solid castings without the use of aluminum provided, of course, mold conditions are correct.

A five-ton heat is made in approximately two hours with a current consumption of five hundred kilowatt hours per ton of charge.

Our experience in the production of steel castings from both acid open-hearth furnaces and acid electric furnaces has convinced us that the electric furnace practice, unless under absolute control, will produce an inferior steel to that of the open-hearth furnace. The time element is much shorter in the electric practice and the chances for abuse of the metal are greater. While proper control is essential in both practices, it is felt that it is of greater importance in the electric practice in order to obtain desirable results.

It is hoped that these suggestions as to the influence of furnace practices in making acid electric steel castings will be helpful to those who may be encountering trouble due to porosity and low ductility in this type of steel.

Written Discussion: By A. W. Lorenz, metallurgist, Bucyrus-Erie Co., South Milwaukee.

The work of Messrs. McCrae and Dowdell seems to confirm the experience of others who have investigated the effect of aluminum additions in cast steel.

Several years ago the writer made a series of tests, the results of which were reported in a brief way before the American Foundrymen's Association in 1928 during a discussion of F. A. Melmoth's paper on "Variables in Steel Foundry Practice." Inasmuch as the present paper deals largely with the effect of aluminum additions, these results are herewith submitted in full, in the hope that they may more fully confirm the findings of Messrs. McCrae and Dowdell.

Our method of procedure involved the casting of twelve test blocks in each of five basic open-hearth heats. Six blocks from each heat were free from aluminum, in accordance with our standard practice on dry sand work. Six received aluminum in the proportion of one ounce per ton. The blocks were cast in two dry-sand molds each containing a set of six blocks. They were poured side by side from a 15-ton bottom-pour ladle. Aluminum was introduced into the aluminum-bearing mold by holding the required amount in the stream while pouring, on the end of an iron rod. This last-minute practice is not a usual procedure in most foundries, and the effect of aluminum is therefore exaggerated to some extent.

The steel, as may be noted in tabulation, was of the pearlitic manganese type. After casting, the blocks were separated into three groups for heat treatment. All blocks of Group I received a simple furnace cooling treatment from 1650 degrees Fahr. All blocks of Group II received

PHYSICAL TESTS ON O. H. STEEL—WITH AND WITHOUT ALUMINUM

Heat	Group	Aluminum	Elastic Limit	Ult. Str.	Elong.	Red.	Charpy
17393	1	With	54950	85500	27.5	39.1	
C .33		With	49000	84700	21.0	27.5x	
		Without	48450	84500	24.0	45.7	
Mn 1.26		Without	51150	85150	23.5	45.7	
	2	With	53450	89400	13.5	14.8x	
		With	62400	90600	24.0	35.0	
		Without	54400	90950	27.5	48.9	
		Without	50950	88150	14.0	18.8x	
	3	With	53450	81200	28.5	46.3	
		With	48400	81000	29.5	45.7	
		Without	54100	81300	32.0	57.6	25.03
		Without	54200	81400	29.5	58.3	25.03
27218	1	With	52700	83850	24.5	33.4	16.76
C .25		With	52750	82850	23.5	35.7	11.19
Mn 1.50		Without	52600	83550	29.5	51.1	15.47
		Without	50000	82300	21.0	41.9x	12.98
	2	With	53900	86300	15.0	20.9x	15.47
		With	63700	88900	24.0	36.6	15.60
		Without	56800	87700	29.5	55.8	20.77
		Without	55550	87050	29.5	58.3	22.16
	3	With	47850	77000	26.5	42.8	14.21
		With	49000	77200	28.5	46.3	15.21
		Without	47500	78700	32.0	63.1	28.30
		Without	48400	80650	30.5	57.6	31.06
27241	1	With	49900	80850	22.0	28.5	
C .28		With	52300	81000	20.5	28.5	
Mn 1.37		Without	50000	82200	25.0	31.5	15.47
		Without	50450	81800	33.0	49.5	15.21
	2	With	55900	84650	17.0	19.5	
		With	56400	85700	15.5	20.2	
		Without	56200	84450	15.5	24.0x	
		Without	55650	83100	11.5	18.1x	
	3	With	46800	75850	28.0	39.4	
		With	48750	76800	24.5	35.7	
		Without	48500	76200	33.0	57.6	20.1
		Without	47650	75100	32.5	57.6	28.3
27206	1	With	47950	81400	21.5	25.8	
C .35		With	48400	80700	22.0	24.0	
Mn .80		Without	48650	82150	27.5	41.9	
		Without	48000	80150	26.0	42.5	
	2	With	54800	86000	21.5	26.1	
		With	54950	86350	20.0	24.4	
		Without	52900	79150	27.5	44.9	
		Without	54050	86250	25.0	34.7	
	3	With	49000	80500	27.0	37.9	
		With	51300	82500	25.5	38.2	
		Without	49200	81350	31.0	58.3	20.77
		Without	47200	81500	30.5	56.8	22.02
27255	1	With	46350	71150	13.0	20.6x	11.19
C .30		With	47750	73350	29.5	30.8	11.78
Mn .77		Without	40150	72700	30.5	49.5	16.76
		Without	40000	71600	32.0	49.7	16.76
	2	With	47700	76250	22.0	26.1	12.02
		With	49550	75950	23.0	27.5	10.83
		Without	49200	77400	31.0	49.5	18.74
		Without	48050	77750	32.0	49.2	18.07
	3	With	44550	73530	25.5	32.8	13.59
		With	42200	73650	24.5	32.1	11.78
		Without	41550	74900	32.5	60.4	23.58
		Without	42300	74100	24.0	41.0	20.77

NOTES: x denotes flaw in bar.

Charpy tests were made on separate bars and should not be mated with tensile results.

Group 1, furnace cooled; group 2, air cooled; group 3, quenched, high draw.

Average Properties of Above Test Bars, With and Without Aluminum

	Elastic Limit	Ult. Str.	Elong.	Red.	Charpy
With Al	51216	81156	22.95%	31.4%	13.30
Without Al	49791	81108	27.45%	47.3%	20.45

an air-cooling or normalizing treatment from 1650 degrees Fahr. All blocks of Group III were heated to 1650 degrees Fahr., water-quenched, and tempered at 1200 degrees Fahr. Each group contained blocks both with and without aluminum. The total number of blocks tested was sixty, of which thirty contained aluminum, and thirty contained none.

The results show that, discarding a few flawed bars, the lowest reduction of area on aluminum-bearing tests was 19.5 per cent, and the highest 46.3 per cent. On the aluminum-free tests, the lowest reduction was 31.5 per cent and the highest 63.1 per cent, with only two tests below 35 per cent. The average reduction was 31.4 per cent for the aluminum-bearing bars and 47.3 per cent for the aluminum-free. Average elongation was 22.95 per cent and 27.45 per cent respectively.

The aluminum bars of Group III (quenched and tempered) showed only a small improvement over the normalized aluminum-free bars of Group II in every case, so that it might be inferred that the full heat-treatment was not of much value. When we inspect the aluminum-treated tests, however, we observe in every heat a very decided improvement with the full quench and temper. This may in a way help to confirm the findings of Messrs. McCrae and Dowdell with reference to the advantage of high tempering treatments, and incidentally to point out why tempering treatments are always advisable.

On the forty-second page of the preprint, reference is made to the trial of higher normalizing temperatures, up to 2000 degrees Fahr., but no results are included on such tests. The use of such temperatures has often been advocated for heavy castings, but their employment for sections approximating test bar size is still open to question. We believe that the full data on this phase of the investigation would be welcome at this time.

Written Discussion: By C. H. Herty, Jr., supervising chemist, metallurgical section, U. S. Bureau of Mines, Pittsburgh.

This exhaustive investigation covers a field which is of interest not only to the manufacturer of steel castings but also to the producer of steel ingots. Many and various alloys are available for purposes of deoxidation at the present time and very little authentic information on their performance has been published. From the results of this paper, it is apparent that if low ductility is due to the presence of Al_2O_3, then either all the deoxidizers used gave types of inclusions similar to Al_2O_3, or all types of inclusions lower the ductility in castings of the type described in the paper. One point not stressed by the authors is the excellent ductility performance of the steel when the Al-Si-Fe alloy was added to the 2½-ton ladle (see Table XIII). It would be of interest to know whether this steel had the same carbon content as the other test pieces in the same table.

Oral Discussion

G. F. Comstock:[1] I would like to call attention to a statement on the bottom of the thirty-fifth page where it says that the ductility of steel castings treated with ferrotitanium was satisfactory and test castings also were

[1]Metallurgical engineer, Titanium Alloy Mfg. Co., Niagara Falls, N. Y.

sound. It seems to me that this fact was not given proper weight in the conclusion of this paper. It is hard for me to see why this is not the answer to the problem if the steel treated with ferro-carbon-titanium showed proper ductility and the castings also were sound. It appears to me that that is what the authors are trying to get. The reason for the different ductility in the steel treated with low carbon ferrotitanium as compared with that treated with ferrocarbon-titanium might be due to the fact that the low carbon ferrotitanium used here contained considerable aluminum. As I understand it, the low-carbon ferrotitanium that is on the market contains 5 to 6 per cent of aluminum. It would seem that that fact would have considerable effect on the results.

C. E. Sims:[2] I want to agree with the authors in regard to the effect of aluminum and its alloys. Aluminum used as an alloy in any composition is equivalent in its effect to pure aluminum. We have also found that other powerful deoxidizers have similar effects in regard to ductility. The effect of aluminum on the ductility of steel works largely through its effect on inclusions. Here is the point where I want to take issue, or at least expand on the statement of Dr. Dowdell. Aluminum increases the yield point of

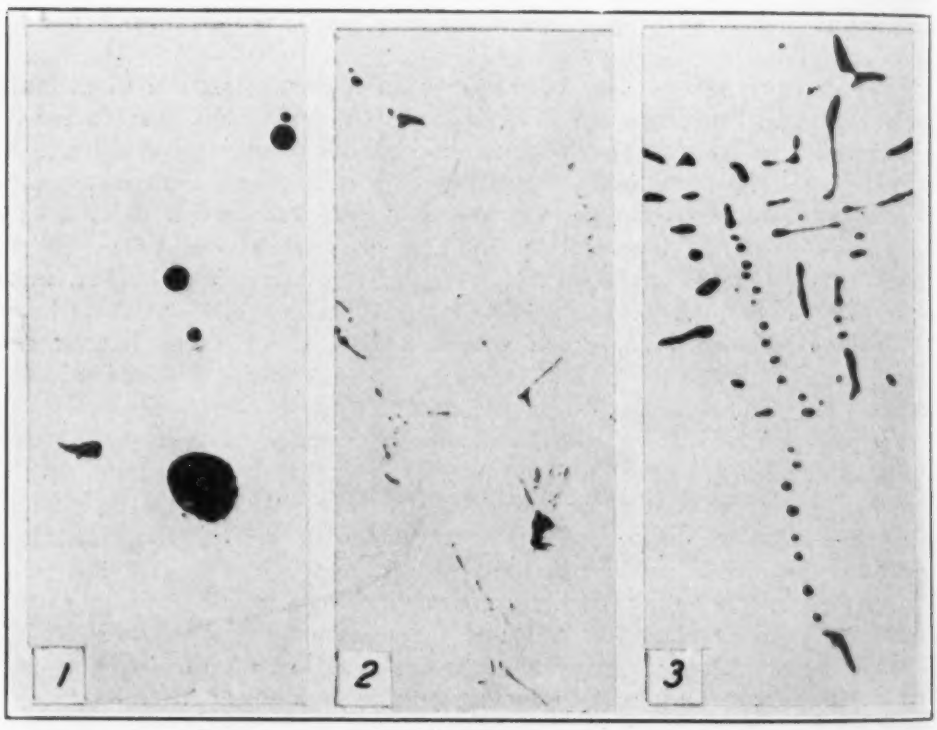


Fig. 1—Spherical Type of Inclusions Characteristic of Steel having High Ductility × 250.

Fig. 2—Small Rounded Inclusions Strung Out in Grain Boundaries of Primary Crystals. Characteristic of the Effect of Aluminum and Accompanied by Low Ductility × 250.

Fig. 3—Similar to Fig. 2 at × 500 to Show the Eutectic-Like Formation of These Inclusions.

[2]Assistant research director, American Steel Foundries, Indiana Harbor, East Chicago.

steel, has little effect on the elongation and a great deal of effect on the reduction of area. From our study of inclusions we can accurately predict whether a steel will have a high ductility or a low ductility merely by looking at the inclusions under the microscope. The type of inclusion has a marked effect on the ductility of the steel. It seems strange that this has been mentioned so seldom before when it is so obvious to a person studying it. Mr. George Comstock spoke of it last fall in Cleveland.[3] When we get a spherical type of inclusion such as iron silicate it occurs indiscriminately scattered through the steel, and whether it has any effect on the ductility or not I cannot say. However, this type does have the very minimum effect on the ductility. This type is illustrated in Fig. I. When we use aluminum we get a different type of inclusion. See Figs. 2 and 3. It is very much smaller. In some cases it is round. In other cases it is very long and narrow, similar to a silicate inclusion which has been rolled out in steel. I am talking of cast steel entirely now. These inclusions are confined to the grain boundaries of the original crystals. They form an envelope surrounding each crystal and they have all the earmarks of a eutectic. See Fig. 3. Our study has not gone far enough to identify the cause of this formation definitely, but it is very apparent that these inclusions are deposited with the last mother liquor to crystallize. The net result, as you can see, is that they form an external phase in the steel, whereas the round inclusions are really inclusions inside the steel. I am sorry I cannot go into greater detail on this. Referring again to the effect of aluminum, we find that it does not always have the same effect on ductility. We have used as high as two pounds of aluminum to a ton of steel and still retained as high as 50 per cent reduction of area. I will only touch lightly on this and say that when a steel is well deoxidized aluminum will ruin the ductility, but if it has plenty of iron oxide in it, in other words, if it is not completely deoxidized, the aluminum will be actually beneficial. It will give soundness in casting without destroying the ductility. I realize that these statements are very general, but I cannot go into detail in this limited time.

Author's Closure

It seems that we did not check up on the analyses, referred to in Dr. Herty's discussion so I cannot say what the check analysis is for carbon. Dr. Herty brought up the point that the presence of all types of inclusions really affected the ductility of castings. I am sure that if Mr. McCrae were here he would agree to the statement that all types of inclusions will lower ductility. I do not see very well how they could raise the ductility. I think most people will agree to that. We did not investigate the various types of inclusions as to their identity, because in this work we wanted a ready solution, if possible, of some quick deoxidizer that was a "cure-all." Analyses were made for both aluminum and for Al_2O_3, by the chemical division of the Bureau on some heats which were deoxidized with plain aluminum. The results showed that if the equivalent of about 1 pound of

[3]George Batty, "Production of Electric Steel for Castings," TRANSACTIONS, American Society for Steel Treating, Vol. 17, 1930, p. 459.

aluminum were added to the large ladle by submerging the aluminum on the rod, and then the ladles were allowed to stand around for a sufficient time, the final steel contained neither aluminum nor Al_2O_3. That would mean of course that an insufficient amount of aluminum was added and with time it had all combined to the oxide. On the other hand when the deoxidation took place in the shank ladles very shortly before pouring, and the additions were made by throwing in aluminum as loose pellets or in the form of rod or anything of that sort, it was found that one-half of the aluminum was lost and unaccounted for in the analysis. The remainder was about equally divided as aluminum and Al_2O_3. That is one of the reasons why we are saying that Al_2O_3 is rather responsible for a large amount of low ductility. In order to insure soundness in steel castings when sand conditions are not ideal, some of this aluminum must be added immediately before pouring. Many steel men will say that sand conditions are ideal and will always add aluminum to the large ladle, but if the sand is not "A 1," they will not have sound castings free of pin holes. In many cases they do not machine the castings to find out whether pin holes exist or not. The reason for not stressing the excellent ductility performance of the bars, 10 and 10A of that table, which were deoxidized with the aluminum-silicon-iron alloy in the 2½-ton ladle is because those values were exceptional. The statement was made, however, that according to "the results as shown by both the data in Table XIII and the appearance of the bars after testing in Fig. 14, were distinctly more favorable to the use of this deoxidizer than were the previous ones." The analyses of the bars 10 and 10a were not checked.

When the steel was deoxidized with the aluminum-silicon-iron alloys in the 2½-ton ladle and allowed to stand for a sufficient time, the chances are that these inclusions had a chance to rise to the top, and the ductility of the final steel was as high or higher with deoxidizers of even plain aluminum than with the aluminum-silicon-iron alloys (Compare Tables 19, 10, 12 and 13).

In regard to the discussion of Mr. Hamilton which was presented by Mr. Sims, there are certainly some points which we ought to correct. Mr. Hamilton mentioned that the results reported do not conclusively show that a higher casting temperature and a higher normalizing temperature are of value. With regard to the higher casting temperature, we had a considerable amount of data from 100 consecutive heats that showed this to be the case. It is regretted that the data had to be deleted from the TRANSACTIONS due to lack of space because the data from 100 heats would have taken about 10 pages of space. However, it seems that a high casting temperature is beneficial. Mr. Hamilton also brought up the question of the data on the seventeenth page, Table 6 as to apparent errors, however, we do not find any apparent errors in the figures. Also, the mechanical properties given in other parts of the paper for steels as low as 0.16 per cent carbon were even quite high in many cases. We did not change the values at all but reported them just as they were so that is why they are so erratic.

It must be admitted that the title of the paper should have included the information that the steel was all made by the acid electric process. In

fact this statement was in the title on two different occasions, but was deleted in order to shorten the title so as to save time for abstractors. As Mr. Hamilton reported, it is possible to make steel by the acid electric method having the properties given in his discussion. We have those same properties in Table 20A which was made toward the close of this investigation but the sand conditions must be ideal if you are going to add all of the aluminum in the large ladle and expect to obtain soundness in the mold.

We are very glad to know that Mr. Lorenz confirms most of the results especially in regard to the lowering of the ductility of the steel with plain aluminum. Both Mr. Lorenz and Mr. Hamilton raised the question about the statement that higher normalizing temperatures in the neighborhood of 2000 degrees Fahr. would be of value. We must admit that we did not have a great deal of information and that it is largely a prediction extrapolated from some of the microstructures. Most of the work was just really a prediction and the statement was put forth somewhat boldly, however, it is interesting to know that everybody does not agree with it. It must be remembered that Mr. Merten is doing some high temperature normalizing on large castings, but when it comes to small test bars it probably is not of a great deal of importance.

Mr. Comstock certainly brought out a very good point, but in no case where the carbon-free-ferrotitanium (thermit process) was used, was the ductility very good. We perhaps have not checked this to as large an extent as possible, but it was checked by the cooperating foundries who said there was no use in trying additional experiments. The main reason was that they had used the thermit type of material, which contained considerable aluminum. I think the point brought up by Mr. Comstock is well worthy of consideration and the ferro-carbon-titanium should be tried at length.

Mr. Sims mentioned that aluminum does not always have the same effect and we believe that it is true. In some cases aluminum is added to the large ladle. You can add any deoxidizer to the ladle, if you give it time enough, and it will cleanse itself if the steel is hot enough but if aluminum is added to a well deoxidized steel immediately before pouring, it seems that it lowers the ductility because it is oxidized by the gases from the mold. We did not determine the types of inclusions because that is a very exhaustive study. However, Dr. Herty and Mr. Comstock have done a large amount of work on that in the past. We do know, however, that the very fine inclusions of Al_2O_3 are very detrimental to ductility.

Finally, in regard to the discussion by Mr. L. E. Gilmore, we are glad to know that Mr. Gilmore agrees with us as to the cause of low ductility and that the initial charge has little effect on its value.

CASTING GUNS BY THE CENTRIFUGAL PROCESS

By Col. T. C. Dickson

Abstract

The mechanical equipment used at the Watertown Arsenal for centrifugally casting guns in chill molds is described. The operation of this equipment and the results obtained when the guns are cast by this method are covered. Several macrographs are included showing the structure at various locations of cast guns. Several tables of physical properties show the results of tests on centrifugally cast guns after heat treatment.

It is concluded that this process not only offers many metallurgical advantages over the ingot-forging-built-up process of gun making but it is also a most economical process.

Casting Apparatus

ONE machine used by Watertown Arsenal for casting guns by the centrifugal process is shown in Fig. 1. A longitudinal cross-section through the rotor, chill mold, pouring box and spout, is shown in Fig. 2. The rotor is a thin-walled steel cylinder which is supported on four rollers, two in each end of the frame. Each top housing contains one roller held in contact with the top element of the rotor by springs to allow for expansion. Each roller is supported by roller bearings provided with forced lubrication. One end of the rotor is connected to a motor. A core case lined with refractory or a cast iron mold is inserted into the rotor, and a flange on the end into which the molten steel is poured is bolted to one end of the rotor to insure rotation of the two as a unit. The interior of the refractory lining or the hole in the chill mold has the shape, generally conical, and the size of the casting required. The end of the mold into which the metal is poured is closed with a cylindrical plate, having a hole into which is inserted the pouring spout through which the metal passes from the pouring box into the rotating mold. The end

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of the mold adjacent to the motor is closed with a plug having an axial hole to permit escape of gas.

A refractory lined mold was used first. The refractory, consisting principally of silica sand, was rammed between a wooden form and the interior of a steel case, the exterior of which fitted closely the interior of the rotor. The form was withdrawn and the refractory dried. The large number of cases and wooden forms and the large capacity of drying ovens required for quantity production, the time required to line the cases and dry the refractory, the time required to remove from the machine the used mold and to insert a new one for each casting, the difficulty of preventing sand from the lining being occluded in the steel and the advantages of quickly cooling the steel in the mold resulted in the exclusive use of chill molds. A single piece of cast iron is bored to the size and shape of the exterior of the gun, with proper allowance for shrinkage and machining, and the exterior surface is machined to fit closely the interior of the rotor. The relatively high speeds used require that the center of gravity of every rotating part shall be on the axis of rotation. The mold is designed to have a chill area five or more times the area of cross-section of the molten steel. While sufficient castings have not been cast in one mold to determine the number of castings obtainable from a mold, it seems reasonable to expect about the same number as from an ingot mold.

Each caliber and model of gun requires a casting that will machine-finish to fixed minimum exterior diameters and length and to a fixed diameter of bore without defects. The exterior and interior diameters fix the thickness of the metal in the wall. For a constant angular velocity of rotation of the mold, centrifugal force varies directly with the radius of the interior cylindrical surface of the molten metal. The rate at which the molten steel enters the mold should decrease as the radius of the interior surface of the metal decreases. The rate of entrance of the molten steel into the mold depends principally on the area of cross-section of the hole in the pouring spout and on the velocity of the metal.

The steel used is made in high frequency induction crucible furnaces which enable the weight required for each casting to be made economically. When a heat is ready for pouring, this type of furnace can be transferred easily to a device that enables the metal to be poured directly from the furnace into the pouring box

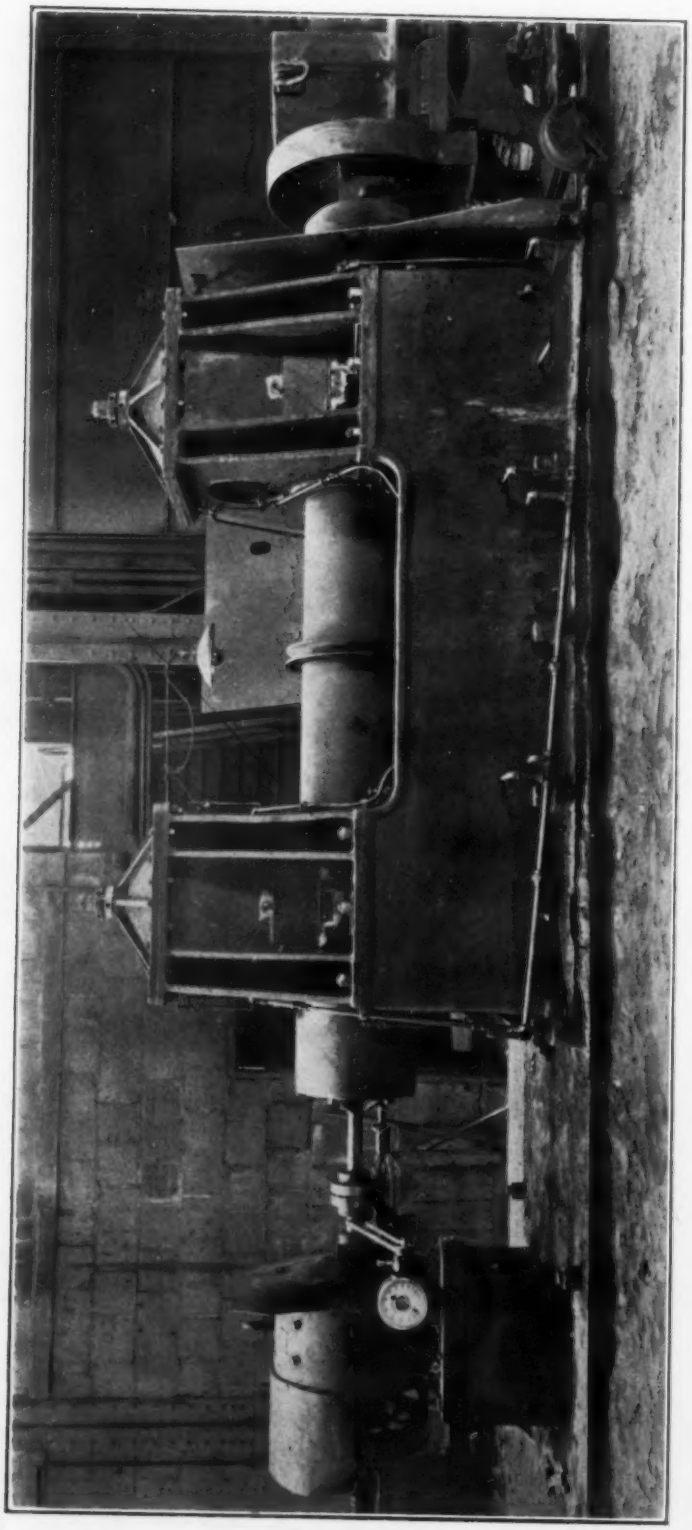


Fig. 1—Photograph of Machine Used for Casting Guns by the Centrifugal Process.

and at the rate desired. The metal should be poured at as low a temperature as will insure the last of the metal to enter the mold being above the solidification point. When practicable, the temperature of the metal is measured with an accurate pyrometer.

Operation

The rotor and mold are brought to the desired speed before pouring of the molten steel is commenced. The speed of rotation may be kept constant during solidification of the steel, or it may be kept constant until the steel is inserted into the mold and then

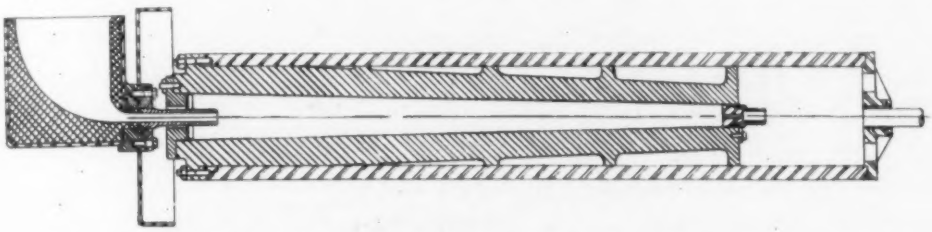


Fig. 2—Longitudinal Section Through Rotor, Chill Mold, Pouring Box and Spout.

gradually reduced, which may be advantageous when the thickness of the wall of the casting is relatively large. If the steel is always poured at approximately the same temperature and other conditions are about the same, the speed of rotation should be fixed by the maximum exterior diameter, the thickness of wall of the casting and the temperature of the mold. The highest speed of rotation with which the exterior surface of castings is free from cracks should be used. The amount of metal that should be poured into the mold to cast a given gun is fixed by the diameter of the cylindrical hole desired in the cold casting that is formed by shrinkage and centrifugal force. The passage of air through the cylindrical bore of the molten metal oxidizes the latter and this is prevented automatically. Rotation of the rotor, without a casting in the mold, maintains a strong draft of air through the mold and cools it. Continuous operation of a machine may require artificial cooling of the exterior of the chill mold.

Solidification in a centrifugal casting proceeds successively from the exterior to the bore. The contraction produced by solidification acts to decrease the diameters of the successive solidifying annuli while the metal is forced outward by centrifugal force. When solidification is complete, the machine is stopped and centrifugal force

ceases to act. Shrinkage cavities should not be formed in the wall of a properly made centrifugal casting. The density of the metal in a centrifugal casting is slightly greater than in a forging of practically the same composition.

Results of Centrifugal Casting

Strain rings show that the metal at the bore of a centrifugally cast gun is under compression, which is not all removed by normalizing, annealing and heat treating. This may be the cause for a centrifugal casting requiring a higher interior pressure to stress the metal at the bore to its proportional limit than required with a forging of the same dimensions and proportional limit. The tension in the exterior metal probably contributes to the formation of exterior cracks. Fig. 3 shows the magnitude of the unit strains.

Chemical analyses were made of thin cylinders, having different radii, cut from the breech, middle and muzzle of a gun cast centrifugally in a chill mold. The percentage content of each element is plotted as ordinates and the distance from the axis of rotation as abscissae. The ladle analysis of each element is shown in the figure. Fig. 4 shows that the carbon content increases appreciably from the exterior towards the bore. Fig. 5 shows a slight increase in manganese towards the bore. The increase in molybdenum is approximately the same as for manganese. Fig. 6 shows the silicon content is fairly uniform. Figs. 7 and 8 show an increase in the phosphorus and sulphur contents towards the bore but the maximum is well within the permissible limits. The higher carbon content at the bore of a centrifugally cast gun makes the proportional limit of the steel appreciably greater at the bore than at the exterior surface. The stresses produced in a gun by the powder pressure are a maximum at the bore and decrease rapidly to the exterior. The segregation of carbon towards the bore caused by the centrifugal process makes the latter ideal for gun making.

Experience indicates that centrifugal castings can be cold-worked to twice the percentage of enlargement of the bore diameter permissible with forgings.

Specifications for Guns

The specifications for gun forgings require not less than 30 per cent of the total weight of each ingot to be discarded from the top

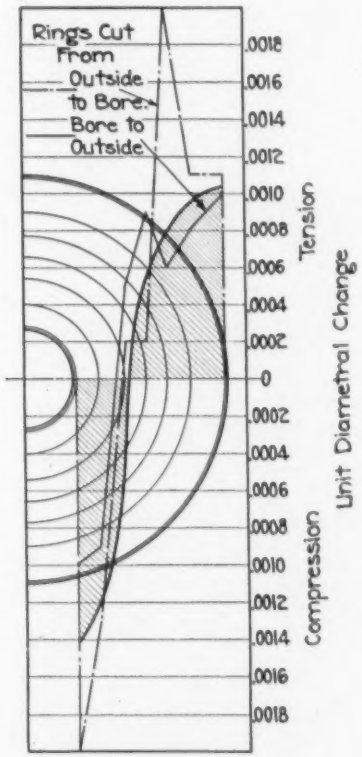


Fig. 3—Curve Showing Unit Strain in Wall of Centrifugal Casting in the As-Cast Condition.

and not less than 5 per cent from the bottom. As the ingot is forged solid, except for hoops and jackets for large calibers, a large amount of metal must be bored out and turned off the rough exterior of each forging. Less than 20 per cent of the weight of the ingots remain in mobile artillery and anti-aircraft guns made by the built-up method. The only discard required from a centrifugal casting is an annular volume adjacent to the bore which contains slag, gas and other nonmetallic inclusions, and which is less than must be removed from a solid forging, and an annular volume adjacent to the exterior surface which may contain slag, cold shuts and shallow cracks. This latter volume does not appreciably exceed the volume that must be removed from the exterior of the rough forging. A radial thickness of about ¾ inch removes all undesirable metal adjacent to the bore. For exterior diameters of about 7 inches, a radial thickness about ½ inch and for exterior diameters of about 4 inches a radial thick-

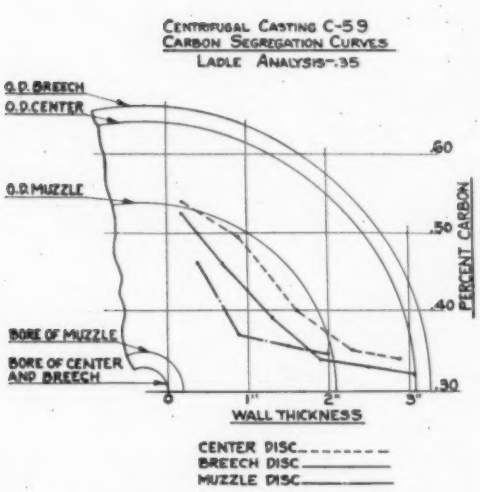


Fig. 4—Carbon Content of Thin Cylinders Cut From Centrifugally Cast Guns.

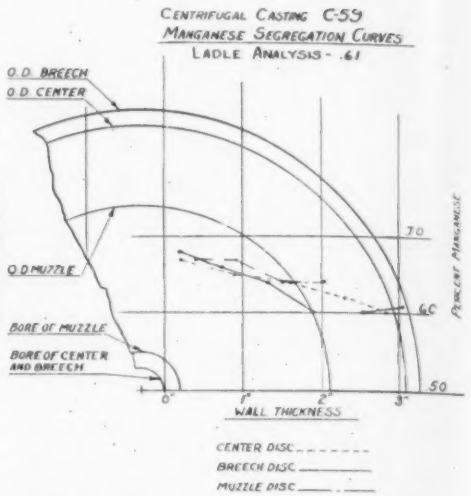


Fig. 5—Manganese Content of Thin Cylinders Cut From Centrifugally Cast Gun.

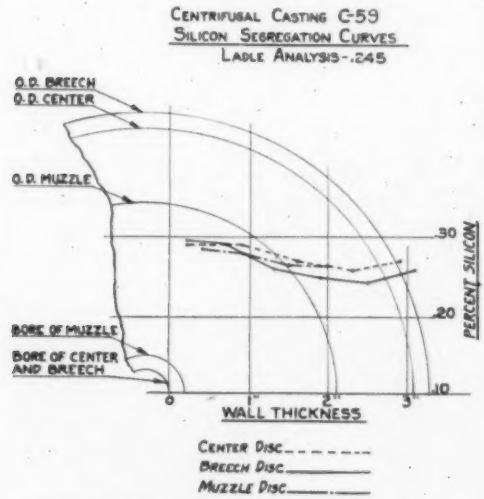


Fig. 6—Silicon Content of Thin Cylinders Cut From Centrifugally Cast Gun.

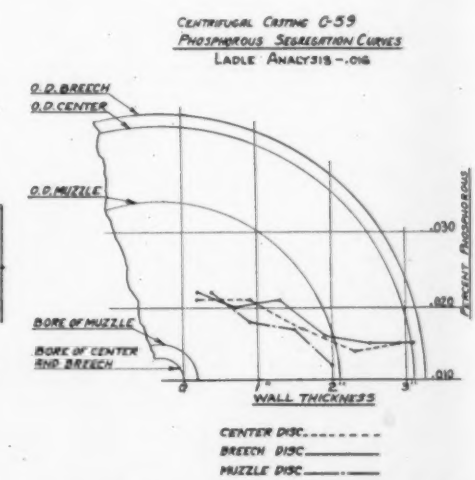


Fig. 7—Phosphorus Content of Thin Cylinders Cut From Centrifugally Cast Gun.

ness of about ¼ inch have proved sufficient to remove roughness, slag, cold shuts and cracks from centrifugal castings.

A mobile artillery gun can be cast centrifugally in one piece in less time than an ingot for either a tube or jacket can be poured, cooled sufficiently and stripped from the mold. The centrifugal casting is removed from the machine immediately after all metal has solidified and when at a temperature above the critical point; it should be inserted into a normalizing furnace to utilize the heat in the casting. The centrifugal process eliminates the operations of heating the ingot for forging and of forging, saves the discard of

at least 35 per cent of the weight of each ingot, and reduces the amount of metal to be bored out.

Prior to the development by Watertown Arsenal of the cold working process, each gun was made by assembling by shrinkage two or more layers of accurately bored and turned forgings. The cold working process enables guns to be made in one piece, exclusive of breech ring and breech mechanism except that high velocity guns

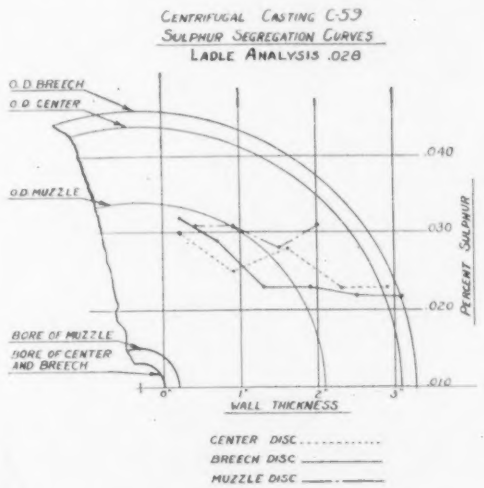


Fig. 8—Sulphur Content of Thin Cylinders Cut From Centrifugally Cast Gun.

are made with a removable liner. Exclusive of breech ring and breech mechanism, guns can be cast centrifugally and cold-worked in about one-third of the time and at about two-thirds of the cost of the ingot-forging-built-up method. This marked reduction in time should prove valuable in an emergency.

Sufficient mobile artillery guns have been cast centrifugally in one piece to establish that the quantity of molten steel required to make one gun by the ingot-forging-built-up method will make from two to three guns of the same size and type by the centrifugally cast cold-worked one piece process. These two processes conserve natural resources.

The experience had to date shows that the output of one machine, with adequate induction crucible furnaces, operating under quantity conditions, should be as follows:

(a) Small sized machine: 37 mm. gun or 75 mm. mortar or 75 mm. howitzer, two per hour.

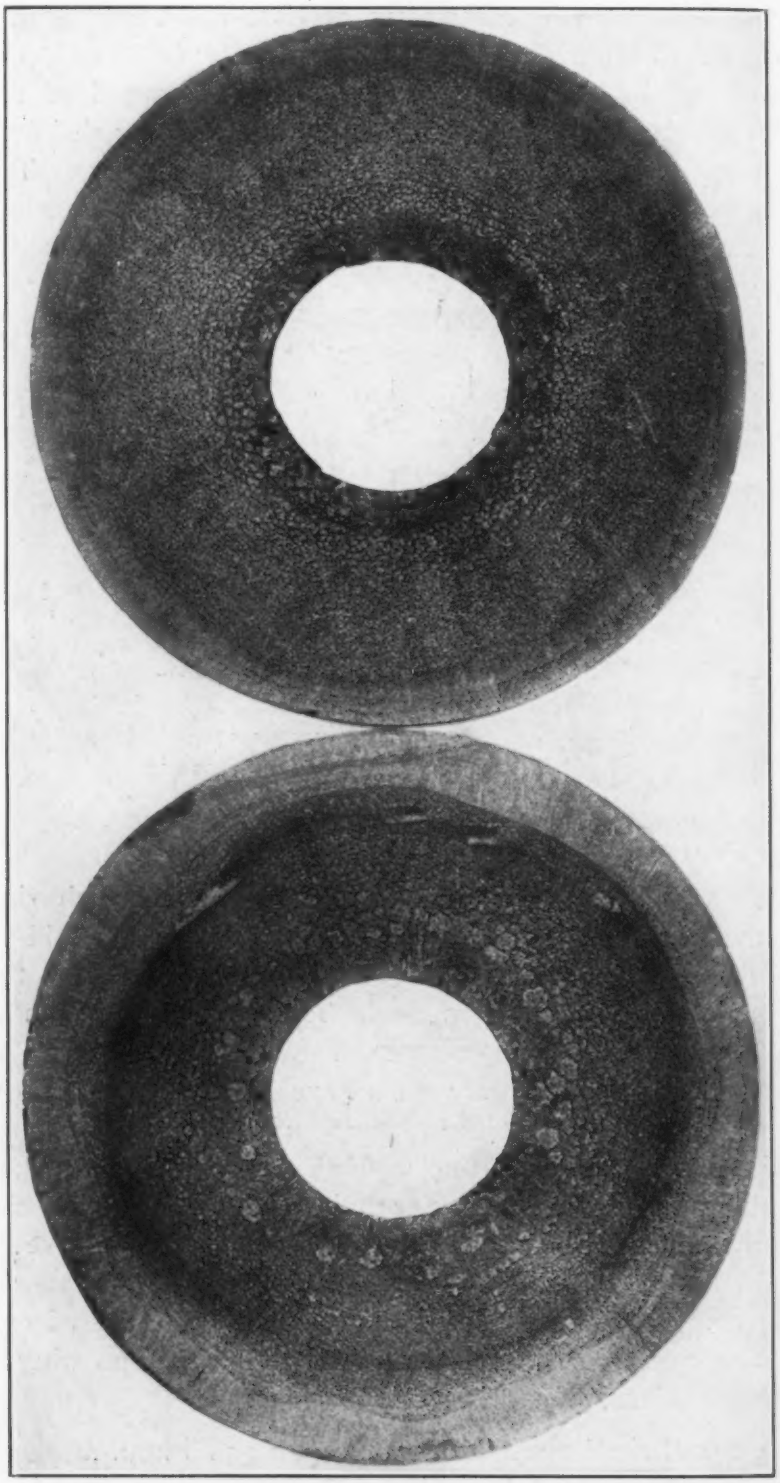


Fig. 9—Photograph Showing Spherical Grains, Indicating that Steel was Poured too Rapidly.

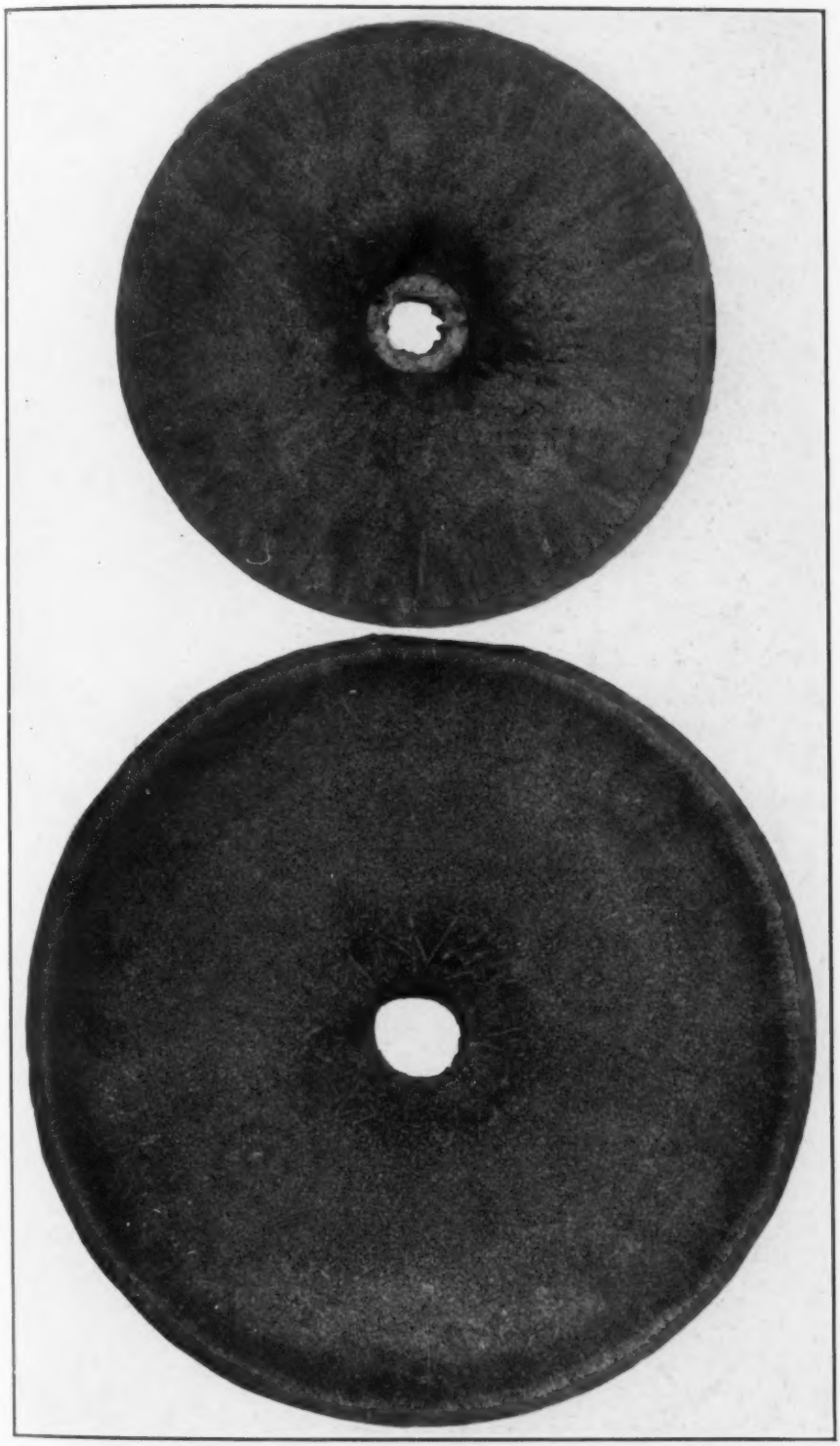


Fig. 10—Photograph Showing Segregation of Slag and Other Impurities.

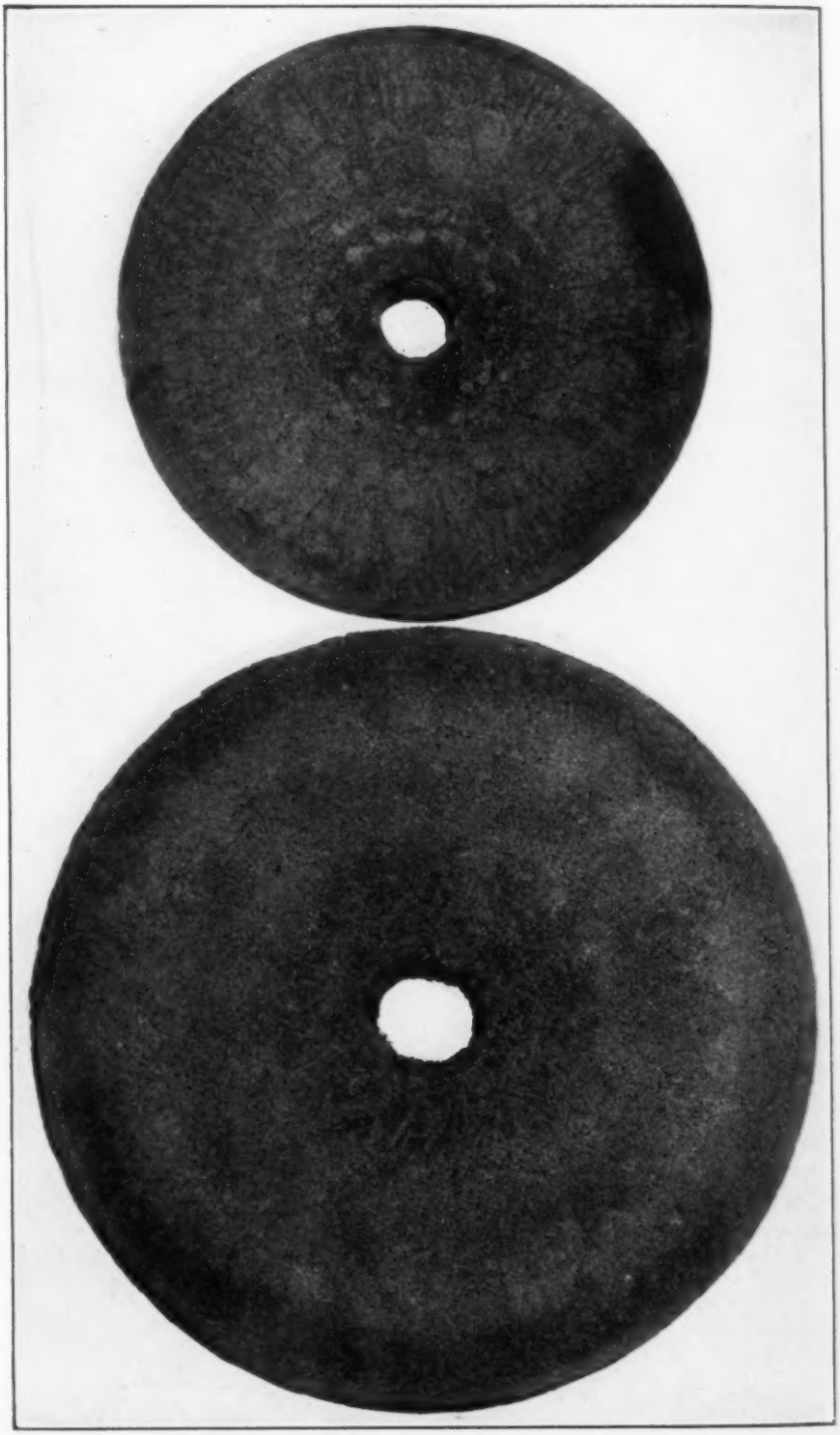


Fig. 11—Photograph Showing Segregation of Slag and Other Impurities.

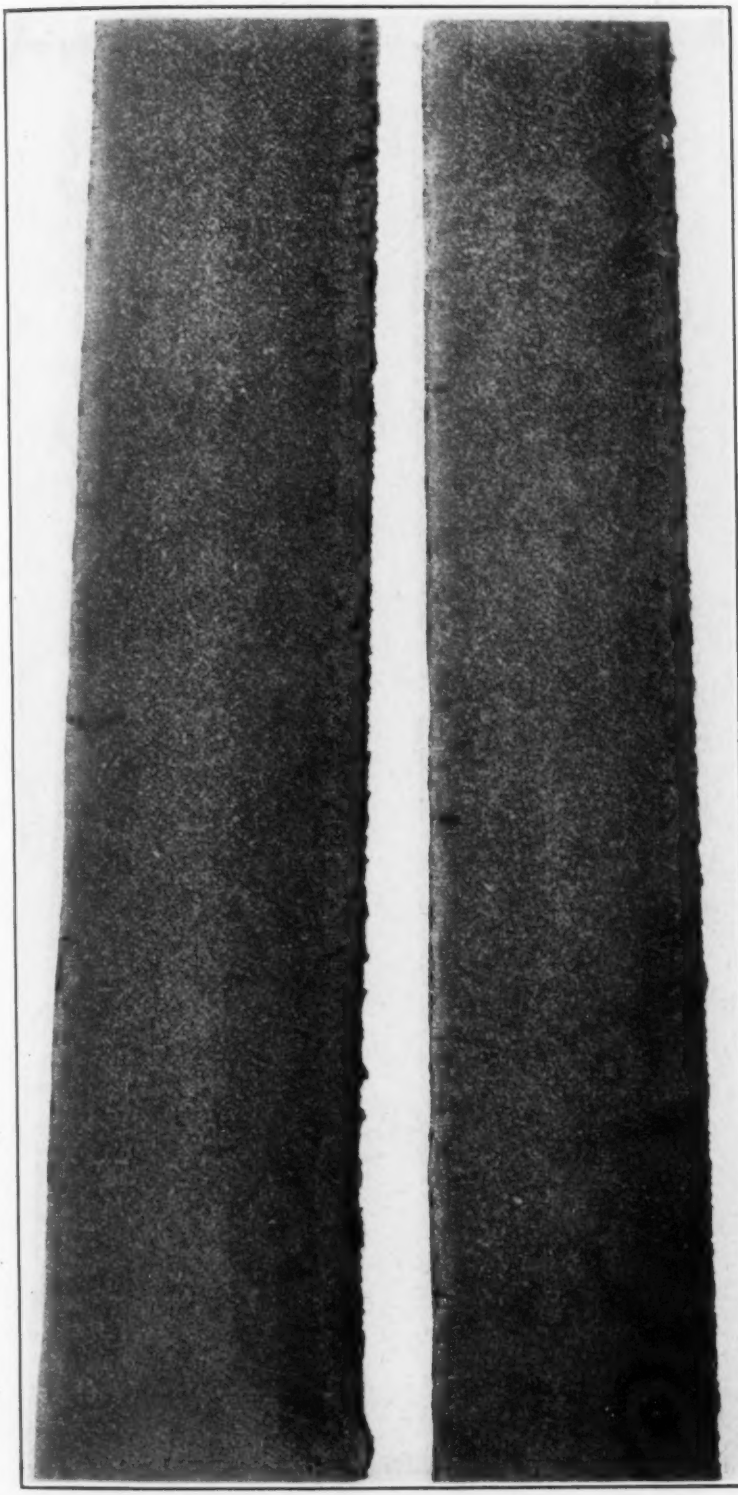


Fig.12—Etched Sections of Full Length Slices Cut from a Centrifugal Casting.

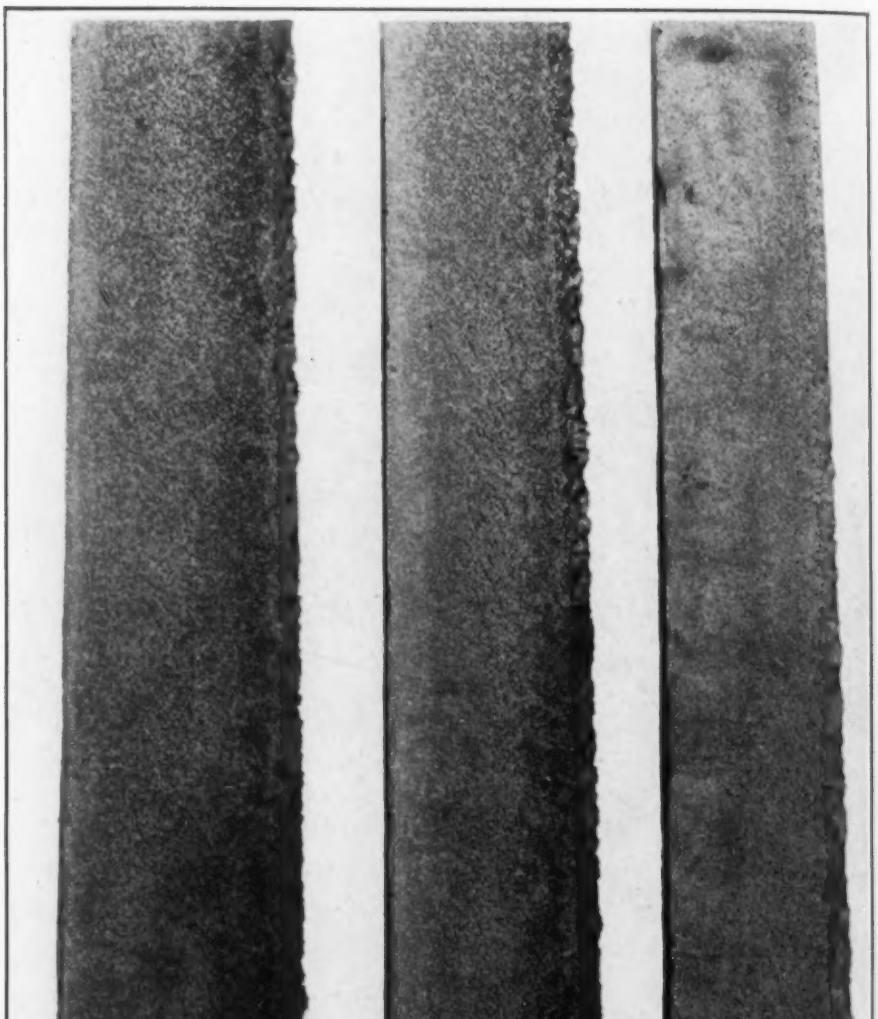


Fig. 12—(Continued).

(b) Medium sized machine: 75 mm. gun or 105 mm. howitzer or liner for 3-inch A. A. Gun, one per hour.

(c) Large sized machine: Tube for 3-inch A. A. Gun or 155 mm. howitzer, one each one and one-half hours.

Metallurgical Considerations

The presence of spherical grains in a centrifugal casting, such as those shown in Fig. 9, indicates the steel was introduced so rapidly into the mold that all of it was not formed into and retained in a cylindrical shape. Probably some of the metal remained flat along the lower part of the hole in the casting and was churned until finally formed into a cylindrical shape. During this churning, many drops probably fell from the metal that was moving upward the cylindrical wall, and such drops appear to thereafter have retained their shape and size. Segregation of slag and other impurities adjacent to the bore are shown in Figs. 10 and 11. The innermost white annular areas in the muzzle sections are practically pure slag. Fig. 12 shows a slice, cut the full length from a centrifugal casting, which slice was cut into five parts for convenience in etching. Near the exterior surface are several shallow cold shuts and cracks. The collection of impurities adjacent to the bore is evident. Test specimens taken so they included the circular lines or thin annuli shown in Fig. 13 and in the muzzle end in Fig. 14 have failed to show that they have a deleterious effect, unless macro-etching reveals an actual separation of the metal, which has not occurred recently. The structures shown in Figs. 14 and 15 are those now normally obtained and give uniform and desirable tensile and Charpy properties. All illustrations showing structures are photographs of the metal as cast and cooled in cinders after a severe macro-etching.

While developing the centrifugal process, several different compositions of steel were tried, and the following is uniformly used: 0.35 to 0.45 per cent carbon, 0.60 to 0.70 per cent manganese, 0.30 per cent molybdenum and 0.05 per cent vanadium.

The suitability of centrifugally cast guns for the military service is being established by subjecting such guns to high hydraulic pressure and to firing tests. A centrifugal casting for a 75 mm. mortar had the following tangential physical properties after heat treatment: air-quenched from 950 degrees Cent. (1740 degrees Fahr.) furnace cooled from 850 degrees Cent. (1360 degrees Fahr.) Water-quenched

from 850 degrees Cent. (1360 degrees Fahr.) furnace cooled from 690 degrees Cent. (1275 degrees Fahr.).

Location	P. L.	T. S.	Elong. Per Cent	Red. Per Cent	Charpy Ft. Lbs.	
Breech	63,000	88,500	28.0	69.2	43.7	39.4
	63,000	90,000	26.0	67.0	43.4	40.5
Muzzle	62,000	91,000	15.0*	47.4	38.4	40.7
	64,000	94,000	21.4*	63.7	45.8	40.5

*Broke near gage mark.

Ladle analysis: 0.32 C., 0.64 Mn., 0.305 Si., 0.015 P., 0.013 S. and 0.48 Mo.

This casting was machined into a cylinder 51.3 inches long, having a bore 2.64 inches and an exterior diameter of 4.204 inches, and was cold-worked without a container. A pressure of 26,250 pounds per square inch enlarged the bore about 0.0007 inch.

Pressure applied in bore	Percentage bore enlargement at distances from muzzle end		
	9 inches	21 inches	39 to 42 inches
35,500	2.7	3.5	3.3
38,000	4.4	5.9	5.4
39,500	6.8	11.8	10.0
40,000	7.4	ruptured	11.7

The maximum enlargement of the bore at 21 inches before rupture was 29.9 per cent.

The metal adjacent to the maximum enlargement, after being soaked at 300 degrees Cent. (570 degrees Fahr.), had these physical properties:

P. L.	T. S.	Elong. in 1 inch Per Cent	Red. Per Cent
96,000	118,000	7.0*	46.4
104,000	114,000	7.0*	54.8
104,000	120,000	24.0*	54.8
108,000	122,000	8.0*	57.4

*Broke at gage mark.

Fig. 16 shows the tube after rupture. Fig. 17 shows the macro-etched structure of three sections after the tube was ruptured.

A centrifugal casting for 75 mm. howitzer, cast in chill mold; heat treatment: air-quenched from 950 degrees Cent. (1740 degrees Fahr.) furnace-cooled from 850 degrees Cent. (1360 degrees Fahr.) water-quenched twice from 850 degrees Cent., furnace cooled from 700 degrees Cent. (1290 degrees Fahr.), had these physical properties:

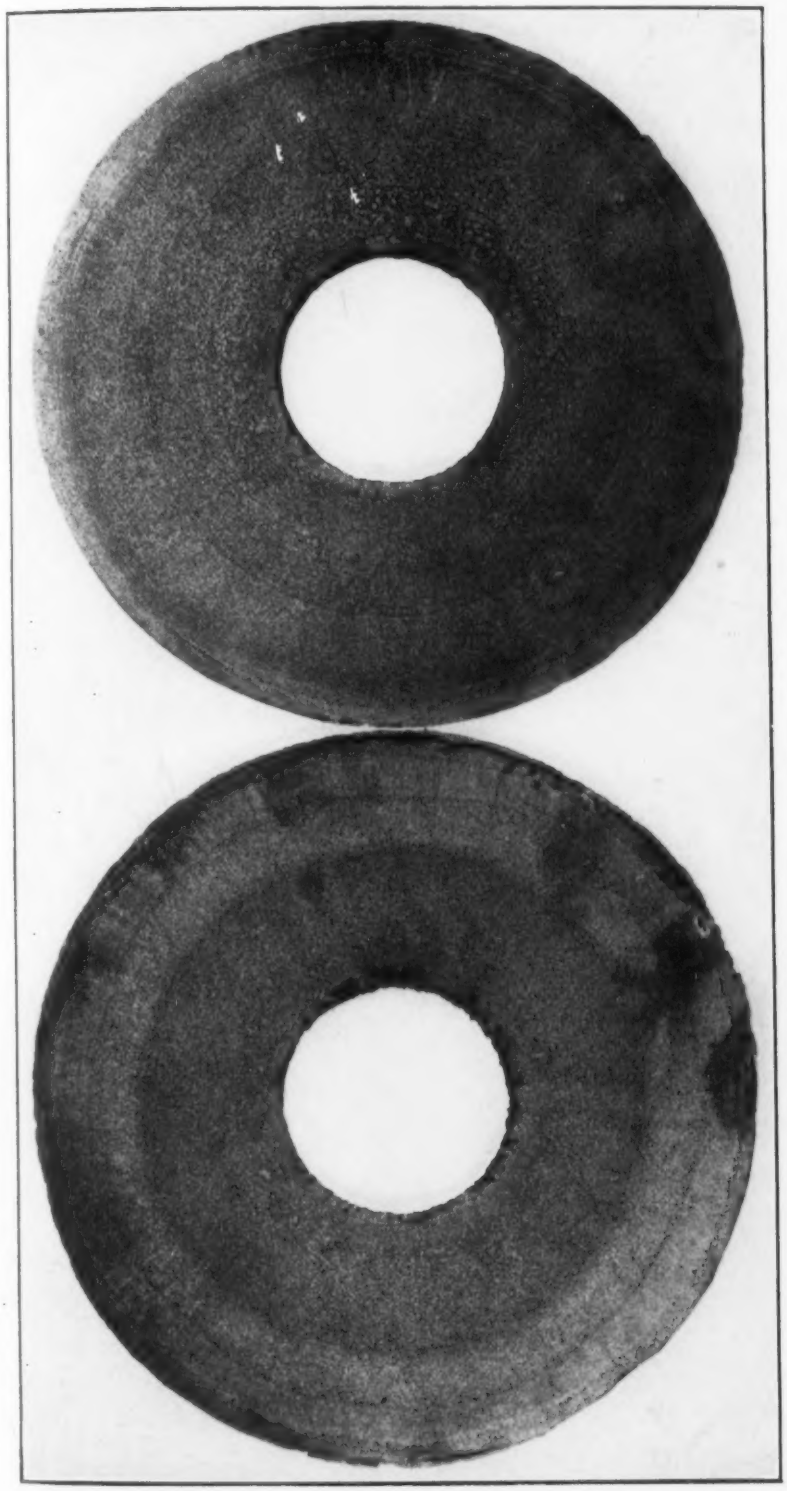


Fig. 13—Photomacrograph of Test Specimen Showing Thin Annuli in Centrifugal Casting.

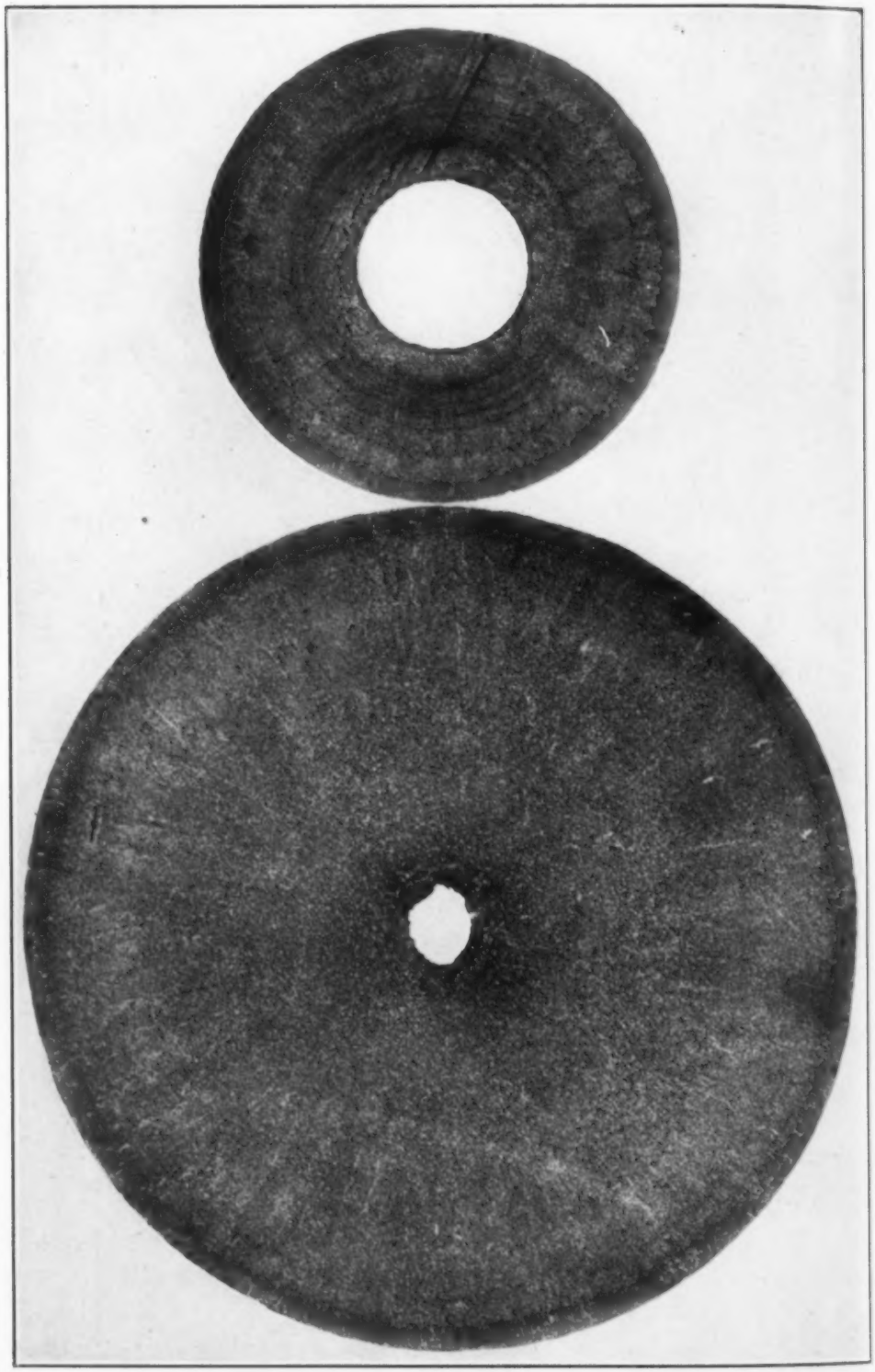


Fig. 14—Thin Annuli of Centrifugally Cast Gun, Muzzle End.

Location	P. L.	T. S.	Elong. Per Cent	Red. Per Cent	Charpy Ft. Lbs.	
Breech	66,000	88,500	20.5*	51.9	38.9	37.4
	69,000	89,000	20.0*	51.9	40.5	37.1
		Diameter of specimen 0.357 inch				
Muzzle	78,000	97,000	20.7*	59.2	32.6	36.6
	76,000	95,000	24.3*	62.0	35.3	39.5

*Broke at gage mark.

Ladle analysis: 0.35 C., 0.58 Mn., 0.29 Si., 0.015 P., 0.017 S., 0.29 Mo., and 0.096 Va.

The casting was bored to a diameter of 2.5 inches and its exterior was machined so the bore could enlarge 40 per cent, or to 3.5 inches, when cold-worked in a container, which was done by applying a pressure of 105,000 pounds per square inch. The bore was enlarged from 37 to 38 per cent between 6 and 27 inches from the breech and from 40 to 42 per cent between 30 and 51 inches from the breech. There are numerous fine checks in the metal adjacent to the bore but their depth appears to be less than the thickness of metal normally left for finish reaming. Not a single crack developed. The section of this casting, at the muzzle end of the powder chamber was soaked at 300 degrees Cent. (570 degrees Fahr.) after cold working and it then had the following physical properties with 0.357 inch diameter of specimens:

P. L.	T. S.	Elong. in 1.4 inches Per Cent	Red. Per Cent	Charpy Ft. Lbs.	
102,000	115,000	11.4*	40.2	29.7	29.8
102,000	112,000	11.4*	51.0	27.4	25.9
106,000	115,000	9.3*	51.7	27.1	25.3
104,000	114,000	15.8*	51.7	29.0	25.2

*Broke near gage mark.

Several forgings have developed serious defects when the bore was enlarged only 6 per cent during cold working. A centrifugally cast gun that will satisfactorily permit a bore enlargement of from 37 to 40 per cent has obvious merits.

A 105 mm. howitzer, cast centrifugally in sand lined mold, had the structure as cast shown in Fig. 18. Heat treatment: air-quenched from 925 degrees Cent. (1700 degrees Fahr., furnace-cooled from 850 degrees Cent., water quenched from 850 degrees Cent. (1360 degrees Fahr.), furnace-cooled from 580 degrees Cent. (1075 degrees Fahr.) Physical properties:

Location	P. L.	T. S.	Elong. Per Cent	Red. Per Cent	Charpy Square Bar	
	70,000	94,200	18.5	30.7	22.2	
	69,000	92,250	16.5	54.6	20.5	
Breech	66,000	89,200	23.5	54.6	32.6	
	70,000	94,100	20.0	43.3	28.0	
Muzzle	63,000	89,300	21.0	34.0	18.1	29.2
	69,000	90,450	16.5	34.0	25.0	38.6

Ladle analysis: 0.22 C., 0.43 Mn., 0.10 Si., 0.017 P., 0.027 S., 0.30 Mo. and 1.16 Ni.

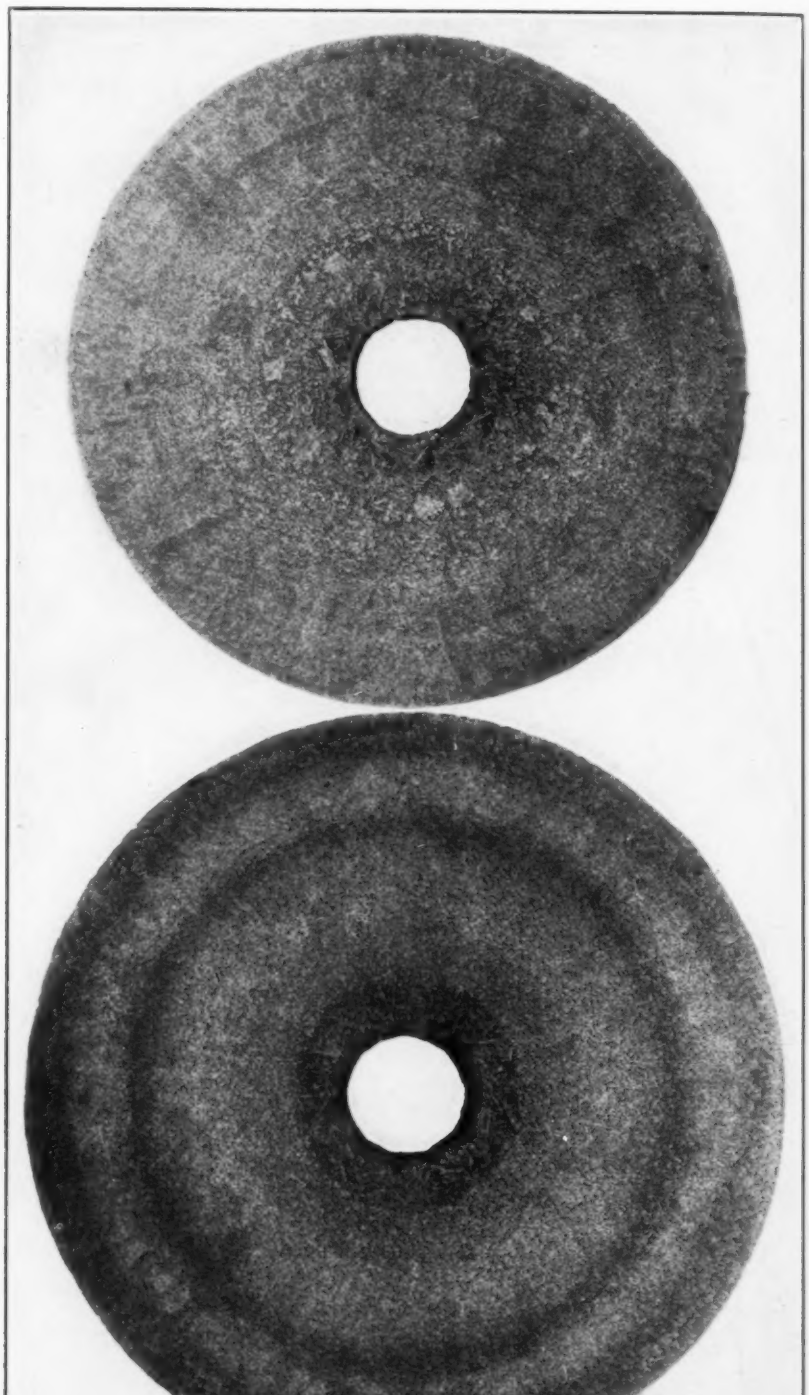


Fig. 15—Photomacrograph of Structure Now Normally Obtained by Centrifugal Casting.

The howitzer was cold-worked to give an average bore enlargement of 7.2 per cent. The service pressure for the howitzer is 28,000 pounds per square inch; it was designed to withstand a pressure of 56,500 pounds per square inch without additional permanent enlargement of the bore. In the firing test, the powder pressure was gradually increased to 55,450 pounds per square inch in the 11th round, with no enlargement of bore. The pressure was 68,800 pounds per square inch in the 12th round which enlarged the front end of the

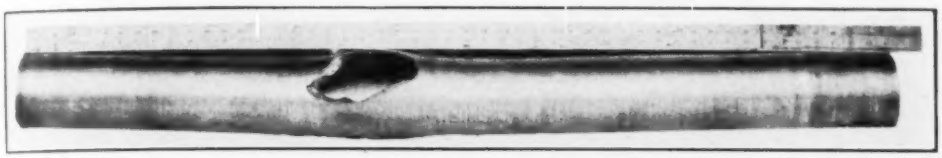


Fig. 16—Photograph of Tube Tested to Rupture.

powder chamber 11.6 per cent without rupture. The pressure in round 13 was 49,800 pounds per square inch, in round 14 was 61,550 pounds per square inch, in round 15 was 61,600 pounds per square inch, after which the maximum enlargement of the powder chamber was 12.06 per cent. A pressure of 68,000 pounds per square inch in round 16 ruptured the howitzer. The enlargement of the bore increased by 8⅞ inches the distance the projectile could be inserted when seated. It appears that this howitzer passed a satisfactory firing test.

A 75 mm. mortar, cast centrifugally in a chill mold, had the structure as cast shown in Fig. 19. Heat treatment: air-quenched from 950 degrees Cent. (1740 degrees Fahr.), furnace cooled from 850 degrees Cent. (1360 degrees Fahr.), water quenched from 825 degrees Cent. (1520 degrees Fahr.), furnace-cooled from 625 degrees Cent. (1160 degrees Fahr.) Physical properties:

Location	P. L.	T. S.	Elong. Per Cent	Red. Per Cent	Charpy Tensile Bar	
Breech	68,000	108,000	14.3	43.6	38.1	22.7
	70,000	109,000	16.3	24.6	15.3	31.8
Muzzle	62,000	100,000	14.3	41.5	38.8	24.7
	64,000	101,000	14.3	54.8	32.7	40.7

Ladle analysis: 0.40 C., 0.70 Mn., 0.265 Si., 0.024 P. and 0.030 S.

The mortar was cold-worked to an average bore enlargement of 6.73 per cent and was soaked at 300 degrees Cent. (570 degrees Fahr.), when its physical properties were:

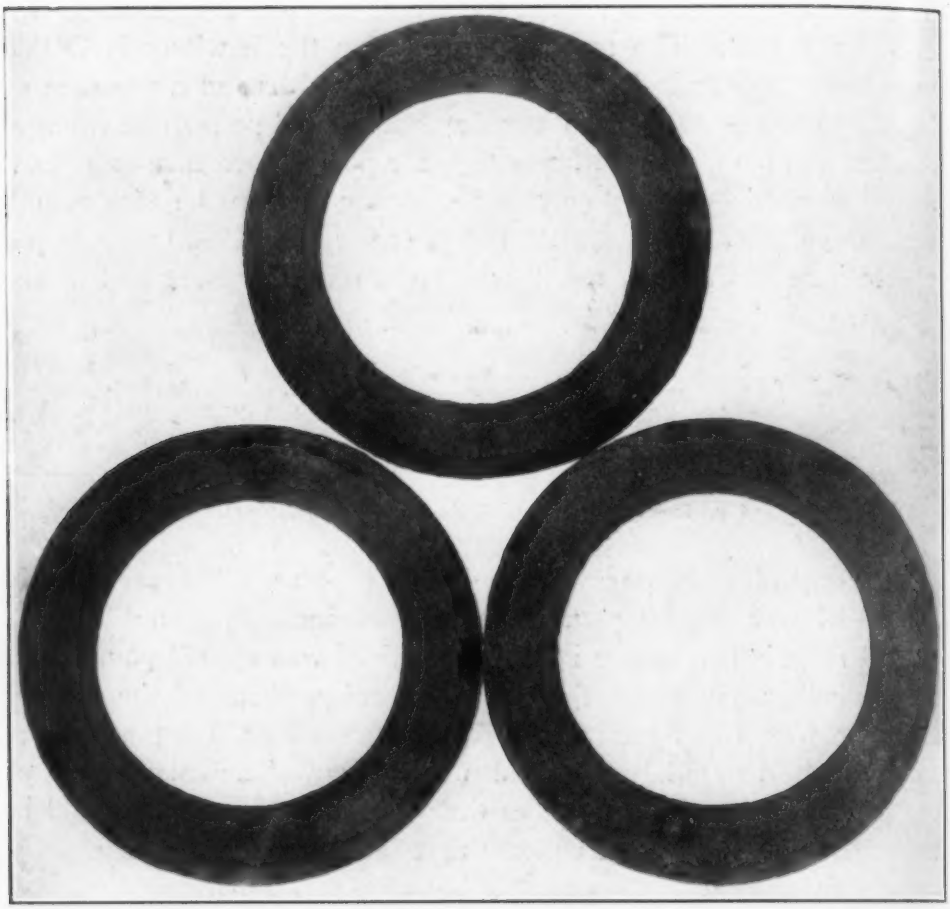


Fig. 17—Photomacrographs of Three Sections of the Ruptured Tube.

Location	P. L.	T. S.	Elong. Per Cent	Red. Per Cent	Charpy Tensile Bar	
Breech	92,000	114,000	5.0	43.4	18.1	19.4
	88,000	104,000	11.0	47.7	19.1	16.7
Muzzle	92,000	116,000	7.0	46.4	14.2	14.7
	96,000	114,000	5.0	36.0	10.6	14.2

The service pressure is 12,000 pounds per square inch and the mortar was designed to withstand a pressure of 26,000 pounds per square inch without additional permanent enlargement of the bore. Three rounds were fired with powder pressures of 26,100, 26,200 and 27,800 pounds per square inch without enlarging the bore. A pressure of 30,050 pounds per square inch enlarged the horizontal diameter 0.001 inch. The pressure was increased to 75,350 pounds per square inch in the 19th round when the maximum enlargement was 17 per cent without a crack or rupture.

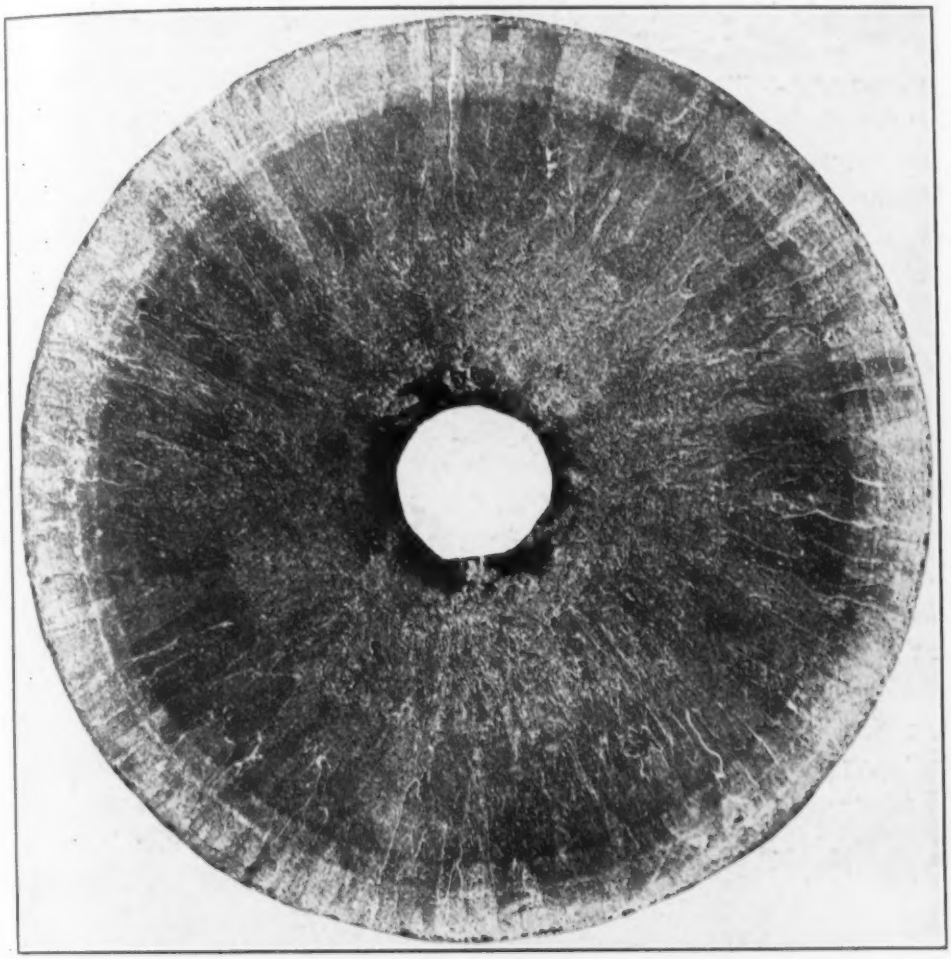


Fig. 18—As-Cast Structure of a 105 Millimeter Howitzer Cast Centrifugally in Sand Lined Mold.

A centrifugal casting, cast in a chill mold, had the structure as cast shown in Fig. 20. Heat treatment: air-quenched from 950 degrees Cent. (1740 degrees Fahr.) furnace-cooled from 850 degrees Cent. (1360 degrees Fahr.), water-quenched from 850 degrees Cent. (1360 degrees Fahr.), furnace-cooled from 675 degrees Cent. (1250 degrees Fahr.). Physical properties:

Location	P. L.	T. S.	Elong. Per Cent	Red. Per Cent	Charpy	Tensile Bar
		Diameter of specimen 0.252 inch				
Breech	60,000	96,000	14.0*	64.8	48.9	42.7
	56,000	92,000	24.0*	64.8	48.9	43.9
Muzzle	60,000	92,000	19.0*	64.8	49.2	54.2
	60,000	92,000	28.0	57.4	46.3	

*Broke at gage mark.

Ladle analysis: 0.38 C., 0.70 Mn., 0.230 Si., 0.013 S., 0.014 P. and 0.31 Mo.

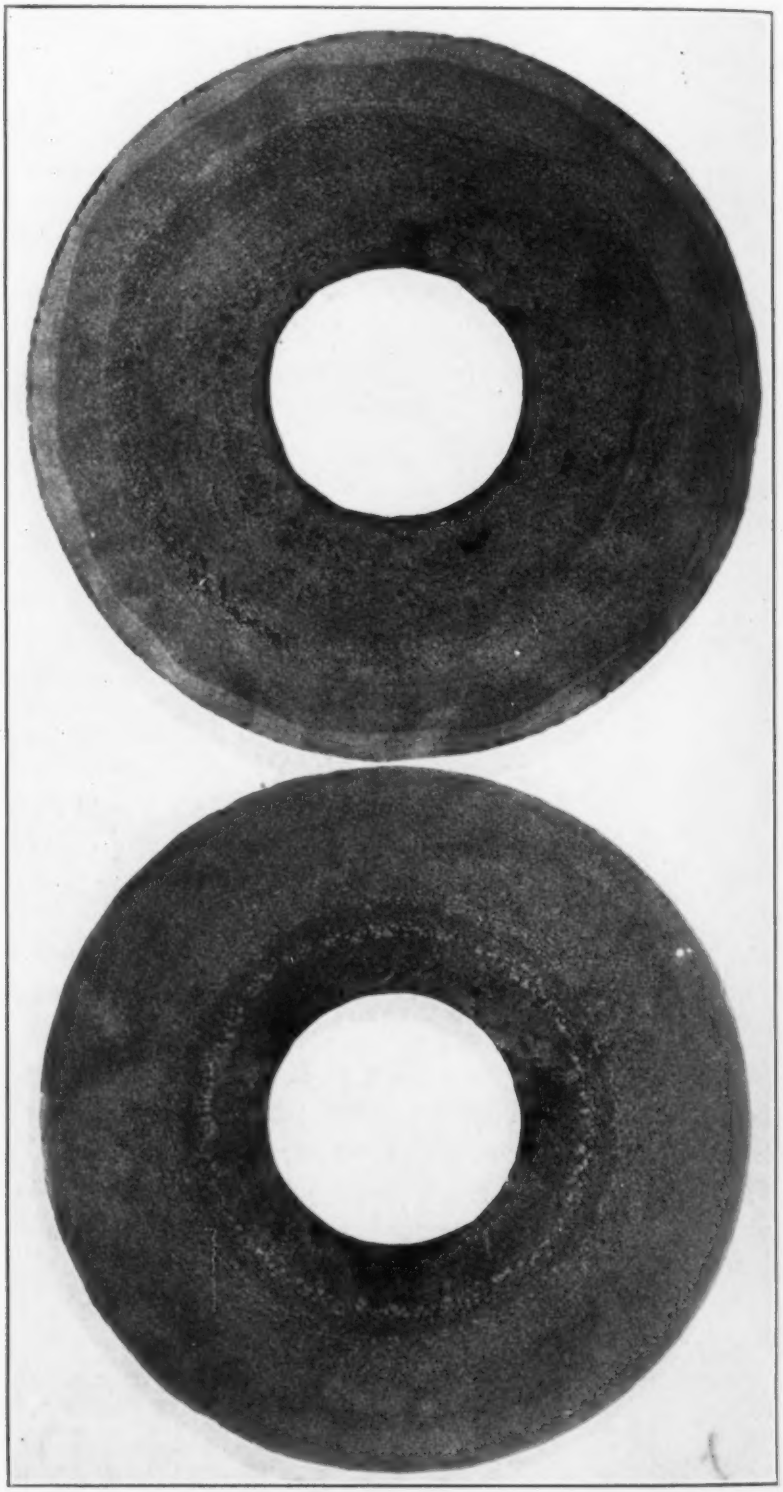


Fig. 19—As-Cast Structure of a 75 Millimeter Mortar Centrifugally Cast in a Chill Mold.

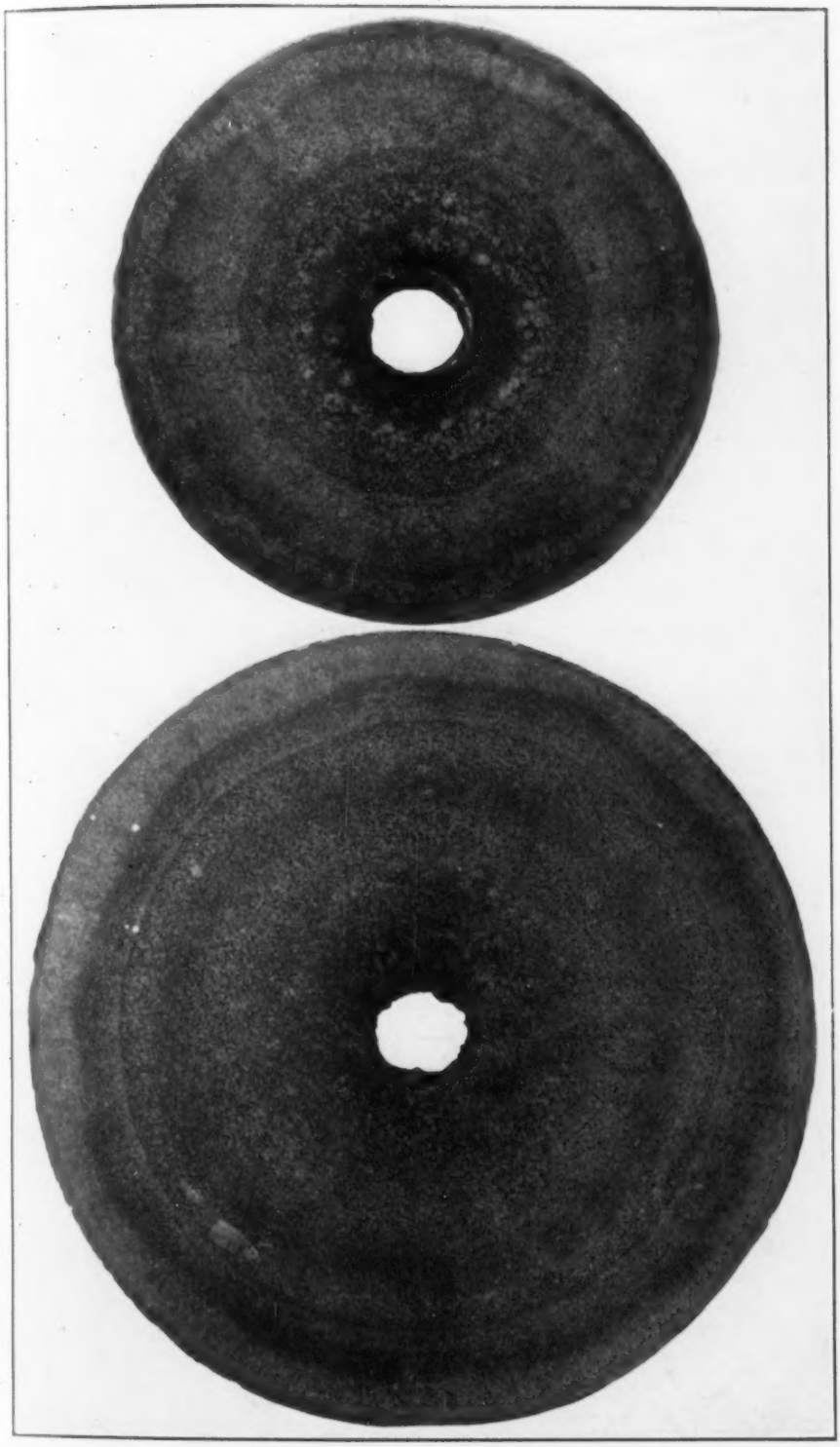


Fig. 20—As-Cast Structure of a Centrifugal Casting, Cast in a Chill Mold.

The casting was machined for a 37 mm. gun, cold-worked to an average bore enlargement of 9.59 per cent, soaked at 300 degrees Cent. (570 degrees Fahr.), and then had these physical properties:

Location	P. L.	T. S.	Elong. Per Cent	Red. Per Cent	Charpy Tensile Bar	
		Diameter of test specimen 0.252 inch				
Breech	88,000	130,000	2.0*	37.2	36.8	35.8
	96,000	128,000	7.0*	46.4	32.0	34.2
		Diameter of specimen 0.16 inch				
Muzzle	105,000	130,000	8.0*	48.5	34.8	33.7
	105,000	130,000	16.0*	35.0	34.0	30.4

*Broke near gage mark.

This casting was made into a smooth bore 37 mm. gun and subjected to firing tests. The service pressure for this gun is 25,000 pounds per square inch. The gun was designed to withstand without additional enlargement of the bore a pressure of 65,200 pounds per square inch. A powder pressure of 93,100 pounds per square inch in 60th round permanently enlarged the bore 0.001 inch; pressures of 102,600 in the 63rd round and of 103,600 pounds per square inch in the 64th increased the enlargement without a crack or rupture.

Conclusions

Advantages of centrifugal castings over forgings for guns:

Metallurgical.

1. Segregation of carbon, which makes the proportional limit of the steel greatest at the bore where the stress is a maximum.

2. Higher tangential tensile and Charpy properties for the same composition.

3. Initial compression of metal adjacent to the bore.

4. Steel is slightly denser, showing that gas, slag and non-metallic inclusions are carried by centrifugal force into the bore or into the metal adjacent to the bore that is removed.

5. Pipe and shrinkage cavities inherent in ingots eliminated.

6. Can be cold-worked to about twice the bore enlargement permissible with forgings which enables the weight of the gun to be reduced and retain the same strength.

Economical.

7. Discard of at least 35 per cent of the weight of each ingot eliminated.

8. Operations of heating for forging and of forging eliminated.

9. Less than half of the quantity of raw materials required to make the same number and kind of guns.

10. Less amount of plant and equipment required to meet war requirements.
11. Time of production greatly shortened.
12. Cost of manufacture reduced.

Acknowledgments

The success attained in developing the casting of guns centrifugally is largely due to the intelligent and interested contributions made by Dr. F. C. Langenberg, Metallurgical Engineer, Captain H. C. Minton, Ordnance Dept., Captain S. B. Ritchie, Ordnance Dept., and Lieutenant Franklin Mitchell, Ordnance Dept.

DISCUSSION

Written Discussion: By H. H. Ashdown, consulting metallurgist and engineer, Y. M. C. A., South Chicago. Without question this is the ideal method for the manufacture of these units and the development of the process reflects great credit on Col. Dickson and all associated with him in this work.

In a process which in so short a period has reached such a high stage of perfection, one feels very diffident to offer criticism. The writer therefore hopes that his remarks will be accepted in a constructive and friendly sense.

The employment of refractory-lined molds, quite apart from heavy labor costs in preparation and attendant risks of free silicate occlusions, are generally bad. Whitworth, in his desire to overcome axial unsoundness by fluid compression, introduced a worse feature in the form of mass segregation throughout his ingots, due to the use of refractory-lined molds.

It is becoming more generally appreciated, the more rapidly steel can be solidified after casting, the more homogeneous and greater will its freedom be from segregation and other inherent defects.

Melting in the induction furnace offers a further advantage for purity, and from the paper it would appear that each gun is cast from an individual furnace charge, which again is another distinct advantage.

Incidentally, if by the usual forging method each small gun was made from a separate ingot both time and money would be saved, as in such, segregation is greatly reduced and a cleaner steel obtained.

It would appear the centrifugal force is sufficient to expel the segregates as they fall out of solution and in Fig. 18 one might reason the long veins of ferrite shown in this illustration are the result of segregates as soon released, dispelling the carbon and then themselves being expelled from the ferrite areas by the rotary force.

As one realizes the limitations to any process, would it be correct to assume, the length of a gun cast by this process, is limited by the flow of the steel before becoming too viscous to form a homogeneous mass?

With the comparatively high carbon content in the bore and the softer

backing, as reported on the seventh page, one can appreciate the high resistance offered to cold work expansion and the ultimate high bursting stress.

It would however be of interest to know if a high carbon steel, or indeed any steel, after being stressed seriously on its working face, beyond its limit of proportionality, is not so surface stressed or ruptured to render it more subject to rapid erosion.

The physical test results from these cold-worked guns, as given on the twenty-first and twenty-fifth pages, are of considerable interest as the reduction of area against the very low elongation appears exceptional, and points to local weakness.

The low and somewhat erratic Charpy test results are not unusual for straight carbon steel, as in that material, both in the normalized and normally treated condition, wide variations are often noticeable.

Without in anyway detracting from the great merits of this process and its marked economy in production, it is essentially a specialty machine, adaptable for a military arsenal.

A private ordnance contractor, in view of the small amount of work he could receive, could not be expected to install such a machine, due to its initial cost, high standing charges and depreciation.

If private capital interests are to be retained in this class of work, to meet emergency calls, it may still be necessary to utilize hammered or pressed forgings, but it is for such manufacturers to devise means for producing the best article their plants are capable of.

Referring to the cost and life of the container, it is questionable if this can approach the average life and cost of a mold. The walls of a mold are unaffected by the molten stream during casting, but in this machine the liquid metal, on each cast, strikes the container in the same wall area and for that reason one would expect local heating and a wash action on the face. This not only would shorten the life of the container, but ultimately cause the tube to "bite in" on the wall and in it, develop contraction stresses.

It would be of interest to learn if, in an instance as illustrated in Fig. 9, where some of the metal has first, in drops, taken a spheroidal form, whether the surface of the spheres had again fused and formed a continuous structure with the matrix. Or whether due to surface oxidation of the spheres they form intersections and initiate points of weakness during subsequent treatment.

As in every operation of steel casting there are occasional failures, now that this process is well established it would be of interest to learn the approximate percentage of failures covering all stages.

G. A. DORNIN:[1] In this gun that you describe, a three-inch gun, what is the relative centrifugal force compared to the force of gravity.

Is there any limit to the size on which this process can be used? Is it limited to small guns? When you get to big guns will other factors enter into it, the same factors that enter into the rupture of the skin of the large ingot?

C. H. HERTY:[2] In the first place I noticed on the casting a pronounced

[1]Metallurgical and consulting engineer, Gathmann Engineering Co., Baltimore.

[2]Supervising chemist, metallurgical section, U. S. Bureau of Mines, Pittsburgh.

running of slag from the center. Did that slag come from slag proper in the induction furnace or from slag which was in the metal of the induction furnace? My second question is—Is the silicon in the metal added at the start of the charge or at the end of the run? My third question may be a little out of order but I have had the question asked me—Is induction furnace steel of a given analysis as clean as electric arc furnace steel of a given analysis, and if not, why not?

A. B. KINZEL:[3] Very often, particularly in the heavy alloys, we have a temperature gradient working with gravity to produce inverse segregation against a cold surface. This is known as the soret effect. It seems to me that the conditions here described would be ideal for its production, in that the force of gravity is replaced by centrifugal force working perpendicular to the cooling surface. I wonder if a small rim, very small indeed, of high carbon material has been noticed at the very outside of the casting immediately around the lower carbon zone.

A. E. R. PETERKA:[4] A man by the name of Leon Cammen, in New York, is making ingots by the centrifugal method. I wonder if we could hear something about this method of centrifugal casting.

COLONEL DICKSON: We know nothing about casting ingots or billets.

MR. RAMSEY: Regarding disposing of the ladle I cannot see how you could pour direct from the furnace into the molds, with the stationary furnace we now have. If any persons have any ideas on that I would like to know.

COLONEL DICKSON: I am opposed to transportation. I do not see why you cannot have the machine located so you can pour your furnace directly into the pouring box.

Author's Closure

To answer Mr. Ashdown: As the length of the gun increases its exterior and interior diameters generally increase which favor the process. To cast relatively long guns it will probably be advisable to introduce the molten metal simultaneously from both ends.

Firing tests made of forgings that have had the bore permanently enlarged by cold working failed to show any appreciable difference in erosion from forgings of the same composition that have not been cold worked.

Macro-etching, microscopic examination and tensile and Charpy test specimens, failed to show that the spheroidal grains or "buttons" in any way affect the strength of the steel and apparently the surfaces of the buttons form a continuous structure with the matrix.

In answer to the remarks of Mr. George A. Dornin, the 75 mm. pack howitzer described when cast at a constant speed of 900 revolutions per minute of the chill mold results in a centrifugal pressure of about 100 lbs. per sq. in. on the exterior surface and of about 10 lbs. per sq. in. on the surface of the hole in the casting. When cast at 1100 revolutions per minute, centrifugal force

[3]Associated with Union Carbide and Carbon Research Laboratories, Long Island City, N. Y.

[4]Metallurgical engineer, Western Electric Co., Chicago.

produces a radial pressure on the exterior of about 150 lbs per sq. in. and at the wall of the hole in the casting of about 15 lbs per sq. in.

155 mm. howitzers have been cast centrifugally. It is not seen why large guns should not be cast by the same process; it seems entirely practicable to design and build a satisfactory machine; as the diameter of the bore increases difficulties of introducing the metal are decreased; in relatively long guns, the molten steel will probably have to be inserted simultaneously into both ends of the chill mold; the larger the exterior diameter, the lower is the speed of rotation to give a desired centrifugal pressure.

In reply to Dr. Herty, may I say that it is believed the slag shown in the illustrations was that on the top of the metal in the induction furnace. All additions, including silicon, are introduced with the charge, except that occasionally further ferrosilicon additions have been made shortly before pouring.

Microscopic examinations and dirt inclusion counts show that the metal in centrifugally cast guns made in the induction furnace is noticeably cleaner than that made in the electric arc furnace by this arsenal. The results obtained by this arsenal indicate that the cleanliness of the metal in guns cast centrifugally of steel made in induction furnaces is approximately determined by the materials that make up the charge.

In response to Mr. Kinzel's query, no chemical analysis has been made of the very thin layer next the exterior of guns cast centrifugally in a chill mold. The analyses made nearest the exterior surface of centrifugal castings indicate a carbon content only slightly greater than the carbon content of the heat and the second analyses nearest the exterior are generally considerably lower in carbon than the later analysis.

SHEET STEEL AND STRIP STEEL FOR AUTOMOBILE BODIES

By Joseph Winlock and George L. Kelley

Abstract

The manufacture of sheet and strip steel and the subsequent forming of these metals by the deep drawing process are not exceptions to the general observation that "the art precedes the science." In the authors' opinion, this is not only because the subject does not easily lend itself to analysis, but also because in the use of empirical methods, many facts which are already known have not been taken into consideration. In this paper are presented a few observations made during the course of a study of the subject from a metallurgical point of view.

Type and Character of the Steel Used

THE steel most widely used for deep drawing purposes is a basic open-hearth steel of the following approximate analysis: carbon under 0.14 per cent, manganese under 0.60 per cent, phosphorus under 0.045 per cent and sulphur under 0.045 per cent.

The maximum carbon content of 0.14 per cent has been specified because experience has shown that an increase in the carbon above this figure leads to a too marked decrease in the ductility of the steel. An exception to this is steel used for light draws, or for a series of light draws with intermediate annealing processes employed to obtain the desired formation. A further exception is steel of the heavier gages (eleven and lower) used in forming certain stampings such as automobile brake drums, axle housings, chassis frames, etc. For the deeper draws of the lighter gaged sheets (18 gage and higher), the present tendency is to lower the maximum carbon to 0.10 per cent, and often to lower the maximum manganese content to 0.30 per cent. No definite conclusions have as yet been reached, however, as to the most desirable analysis.

Alloy steels have not been extensively used except in special cases, partly on account of the added cost, and partly because the in-

A paper presented before the Semi-Annual meeting of the society held in New York City Feb. 7 and 8, 1930. The authors, members of the society, are metallurgists of the Edward G. Budd Manufacturing Co., Philadelphia. Manuscript received Nov. 25, 1929.

herent strength of the steel is generally of secondary importance. Strength and rigidity are usually imparted to a stamping by designing it so that it will most effectively resist the forces imposed upon it in use by virtue of a high section modulus. For example, in Fig. 1 are pictured two pieces of steel alike in every respect except that one has been formed into a channel section as shown. If a force, increasing in magnitude, is imposed upon each of these pieces of

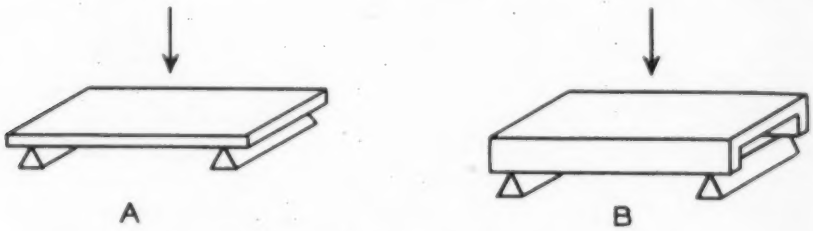


Fig. 1—Diagram Showing General Method of Imparting Strength and Rigidity to a Stamping.

steel in the direction indicated by the arrows, it will be found that a very much smaller total force is required to deflect the rectangular section (a) than the channel section (b).

Steel of the above analysis is particularly susceptible to conditions which tend to produce a lack of uniformity in the finished sheet or strip. As has been indicated in a paper by the authors[1], the physical properties of finished sheets may often show a wide variation not only between individual sheets of the same lot, but also in the same sheet. Impurities are more apt to segregate in the ingot of steels of this type than in steels of a higher carbon content, and it is difficult to obtain satisfactory deoxidation of the molten steel. Blowholes are more apt to form in steel of this carbon content on account of its relatively low dissolving power for gases at low temperatures, and the use of too much aluminum to overcome this condition results in a "dirty" steel. Subcutaneous blowholes and those formed near the center of the ingot, being less liable to oxidize than those on the surface, will, however, usually weld together satisfactorily in the subsequent rolling processes.[2] Unwelded blowholes together with "pipe" are the chief causes of "laminations" in sheet and strip steel.

Steel of this analysis, particularly when rolled to light gages, is

[1]Winlock and Kelley, "Testing Automobile Sheet Steel," TRANSACTIONS, American Society for Steel Treating, Vol. 12, Oct. 1927, p. 635.

[2]Stead: "Notes on the Welding up of Blow Holes and Cavities in Steel Ingots." *Journal*, Iron and Steel Institute. 1911, No. 1, p. 54 and 1912, No. 1, p. 104. See also Brearly: "The Welding of Steel in Relation to the Occurrence of Pipe, Blow Holes, and Segregates in Ingots." *Journal*, Iron and Steel Institute. 1921, No. 1, p. 27.

especially susceptible to large grain size changes occurring in the various annealing operations.

Manufacture of Sheet Steel

There are two principal processes for rolling steel from the ingot to the sheet metal used for deep drawing purposes: (1) the sheet steel process and (2) the strip steel process. The essential operations, i.e., hot rolling, cold rolling, annealing, and pickling to remove scale are included in both, but the number and sequence of these operations together with the mill equipment and methods of carrying them out differ considerably. There may also be differences in each process according to variations in individual mill practice.

In the manufacture of sheet steel, the ingot is first rolled to a bloom, and then, by further rolling, to the different shapes. The final product of this series of hot rollings is the "sheet bar" which measures approximately eight inches in width and is of a thickness which will give the desired weight per linear foot. The weights per linear foot of the sheet bars vary with the length and gage of the sheets to be subsequently rolled. The sheet bars are cut to a length corresponding to the approximate final width of the sheet.

The bars are then often pickled to remove scale and *cross-rolled* to approximately one half the length of the finished sheet. The product of this rolling is called a "breakdown." (Some types of sheet are hot-rolled to the finished length immediately, but more often a pickling operation is introduced at this point.)

After reheating, the sheets are rolled in packs of two, three, or four (depending upon the gage) to the final length. Following this, and either before or after being pickled to remove scale, the sheets are annealed. They are then rolled separately on the cold rolls and given a further annealing. This is usually followed by an additional cold rolling, and in some instances, the sheets undergo still another annealing operation.

Manufacture of Strip Steel

In the manufacture of strip steel, the ingot is first rolled to a slab or billet on the hot mills, cut to the proper length and, after reheating, rolled *in the same direction* on hot mills (usually set up in tandem) to the desired thickness. The strips are next either coiled immediately or are allowed to cool and then wound into coils. These

coils are annealed, pickled to remove scale, and then cold-rolled, followed by another annealing and often a final cold rolling.

The number and sequence of mill operations in the manufacture of both sheet and strip steel are governed not only by the deep drawing qualities and dimensions required in the finished sheet or strip, but also, in a greater degree, by the quality of the surface desired. Sheet and strip steel may be designated "hot-rolled," for example, and used in those stampings where the surface requirements are not high.

In considering the foregoing, it may be seen that a metallurgical study of the manufacture of sheet steel and strip steel, and the forming of these metals by the deep drawing process involves principally an analysis of the effect on the structure and physical properties resulting from hot rolling in the range of temperatures employed, the amount of the reductions produced on the hot and cold mills, and the changes which take place as a result of the different annealing operations, and, finally, the effects on the steel of the cold deformation produced by deep drawing operations.

Hot Work

In Fig. 2 is depicted graphically the well-known changes which occur in a steel containing about 0.10 per cent carbon while the metal which is being hot-rolled cools from above the critical range to atmospheric temperature. Sheet bars are not usually heated as high as the Ac_3 point before rolling, but slabs and billets, on account of the greater reductions necessary, are usually heated well above the critical range.

Above the point B which represents the Ar_3 point [about[3] 1560 degrees Fahr. (850 degrees Cent.)], the metal exists as a solid solution of carbon in gamma iron. In cooling through the Ar_3 point, an allotropic change takes place and ferrite begins to be generated in the austenite as alpha iron.* This phenomenon proceeds along the line AO until the temperature designated by the point D (the Ar_1 point) is reached, when the austenite, now of eutectoid composition, changes into pearlite.

[3]The exact temperatures cannot be given because of the retardation of the transformations which occur in direct proportion to the rate of cooling.

*The possible change of gamma iron to beta iron occurring at this point and transforming at the Ar_2 point [about 1420 degrees Fahr. (770 degrees Cent.)] has been omitted from this description for the sake of clearness.

In a steel containing 0.10 per cent carbon which has been cooled slowly, there is only the small amount of 11.7 per cent pearlite, and, as pearlite contains ferrite, there is only the small amount of 1.5 per cent cementite. Sometimes only traces of cementite or pearlite may be seen under the microscope because, as Sauveur and Krivobok report, alpha iron is capable, after slow cooling, of retaining in solution at least 0.06 per cent carbon.

The temperature reached in the sheet bars, billets, and slabs in heating preliminary to hot rolling depends largely on the gage desired.

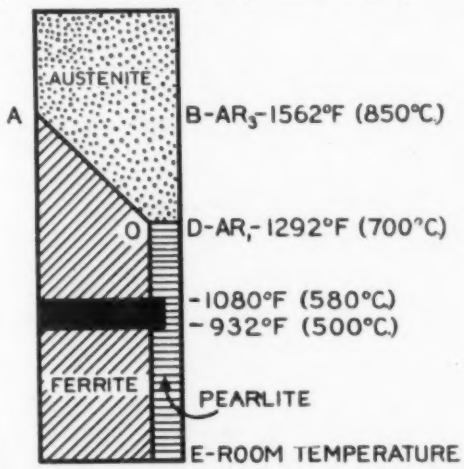


Fig. 2—Diagram Showing the Changes Which Occur in a Steel Containing About 0.10 Per Cent Carbon as it Cools, While Being Hot Rolled, to Atmospheric Temperature.

The temperatures are kept as low as possible to prevent excessive scaling, but at the same time high enough to afford the necessary time for rolling while the metal is sufficiently plastic. In most cases, however, the temperatures reached are above 1450 degrees Fahr. (788 degrees Cent.).

As the rolling commences, the crystalline grains of the metal are drawn out in the direction of rolling, but due to the high temperature, "spontaneous" annealing takes place, and because undisturbed cooling is necessary for large grain growth to occur, a refining of the crystalline grains takes place.

In the temperature range denoted by the line DE, the steel consists of an aggregate of ferrite and pearlite. Permanent deformation of the pearlite occurs as soon as the Ar_1 point is passed, but distortion of the free ferrite does not take place on account of the concomitant annealing, until the temperature zone indicated by the heavy

black line, about 1080-930 degrees Fahr. (580-500 degrees Cent.) is reached. This range of temperature may be designated as the demarcation between so-called "hot working" and "cold working."[4] The temperature necessary to bring about spontaneous annealing in this range decreases as the amount of the reduction increases. At about 930 degrees Fahr. (500 degrees Cent.), which is the bottom of the range, the atomic mobility becomes so low that spontaneous annealing cannot take place. It follows from this, that little, if any, "strain" or "work-hardening" takes place in the metal if the steel is finished on the hot mill above this temperature.

By continuing the heavy black line into the pearlitic area, an attempt has been made to show graphically that the ferrite in the pearlite is also affected by cold work in much the same manner as is the free ferrite.

Cold rolling followed by heating to this recrystallization range probably causes a greater ease of rearrangement of the particles which aids in the spheroidization of the cementite by surface tension. This is the main reason, the authors believe, for the claim that spheroidization of the cementite takes place at a faster rate when the steel has been strained.[5]

As is well known, steel in the spheroidized condition is more ductile than when in the pearlitic condition.[6] Fig. 3 shows the microstructure of a specimen of sheet steel in which the cementite is completely spheroidized.

Stead,[7] Sauveur,[8] and others have shown that phosphorus and dendritic segregation are contributing causes for the banding of the ferrite and pearlite often seen in many steels. The authors would like to suggest as a possibility an additional cause, in certain cases of this type of segregation, which is based on the effect of rolling steel in the temperature range shown by the line BD in Fig. 2. During this cooling from B to D, as has been pointed out, crystalline grains of ferrite and austenite exist which, while in contact with the rolls,

[4]Sauveur and Lee, "The Influence of Strain and of Heat on the Hardness of Iron and Steel," *Journal*, Iron and Steel Institute, No. 2, 1925, p. 329.

[5]Whitely, "The Effect of Cold Work on the Divorce of Pearlite." *Journal*, Iron and Steel Institute, No. 1, 1918, p. 353. See also discussions by Rosenhain and Desch.

[6]Sauveur, "The Metallography and Heat Treatment of Iron and Steel." (Third Edition) p. 286.

[7]"Iron, Carbon, and Phosphorus," *Journal*, Iron and Steel Institute, No. 1, 1915, p. 140.

[8]"Crystallization of Iron and its Alloys." TRANSACTIONS, American Society for Steel Treating, Vol. 4, 1923, p. 12.

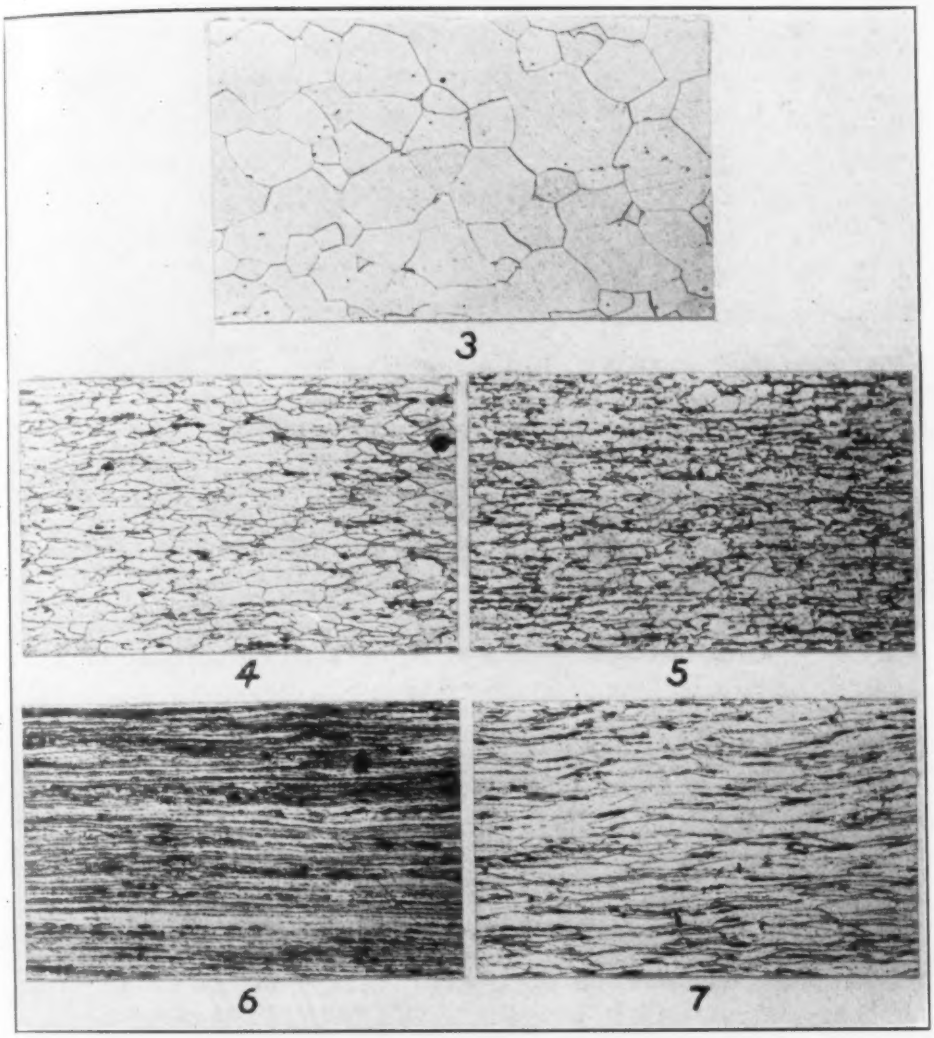


Fig. 3—Cross Section of a Specimen of 0.08 Per Cent Carbon Sheet Steel Showing Spheroidized Cementite. × 75.
Figs. 4 to 7—Showing Different Microstructures Which May Be Obtained in Producing Hot-Rolled Sheet Steel. × 75.

are elongated and drawn out in the direction of rolling. It is postulated that the rejection of the ferrite to the boundaries of the austenite[9] while it is in this elongated condition, and constantly changing in ferrite concentration as the temperature falls, causes a mechanical separation of the constituents resulting in a banded structure. This suggestion seems to be substantiated by the fact that some

[9]Howe: "The Metallography of Steel and Cast Iron." p. 170 et seq. McGraw-Hill Book Co., Inc.

sheet and strip steels are freed from this heterogeneous structure by a relatively short heating above the Ac_3 point.

In the case of sheet steel of the lighter gages and strip steel of the narrower widths, the finishing temperatures on the hot mill are usually so far below the temperature at which spontaneous annealing takes place that a considerable amount of the deformation is actually cold work. This accounts for the fact that steel which has been cold-rolled on the hot mill is particularly susceptible to grain size changes on subsequent annealing or heating for further rolling. Low finishing temperatures are usually necessary, however, in order to obtain good surface qualities. In Figs. 4 to 7 are shown some of the different microstructures obtained in hot rolling.

The amount of cold work in steel rolled on the hot mill may often be so great that "exaggerated" grain growth may occur on annealing and may persist through to the finished sheet. This is unlikely to occur in strip steel, however, because the amount of reduction produced by cold rolling is usually so great that all vestiges of large grain growth disappear in the annealing. Hot-rolled sheet steel of the heavier gages (14 and under) and hot-rolled strip steel of the greater widths (30 inches and over) by virtue of their greater thickness and consequent less rapid rate of cooling, usually show very little of the effects of cold work.

The wider the sheet or strip and the lighter the gage being rolled, the greater is the tendency toward nonuniform reduction in hot rolling. This is because of the high working pressures, and because the rolls often lose their shape on account of expansion and contraction. Sheet bars rolled on rolls which are too convex result in sheets being produced which are of a lighter gage in the middle than on the edges, and if rolled on rolls which are too "hollow," the reverse condition results. The tendency toward nonuniform reduction is accentuated if the sheet bars, billets, or slabs have not been uniformly heated. Rolls are ground slightly concave in order to compensate for these conditions as much as possible. When subsequently cold-rolled, parts of such sheets will of necessity receive unequal amounts of cold work. It is considered possible that large grain growth might occur when sheets in this condition are annealed at low temperatures because of the presence of excessively large strain gradients.

Long heating at high temperatures preparatory to hot rolling is injurious because of the pitting of the surfaces of the metal by scale.

Decarburization of the metal will also often take place in heating unless a reducing or neutral atmosphere exists in the furnace. Decarburization, obviously, detracts from the homogeneity of the steel. An additional cause of a lower carbon content on the surfaces than in the middle of the metal is suggested by one of Howe's experiments which shows the effect of the rejection of ferrite by the austenite as the steel cools through the critical range.[10] In this experiment, a specimen of hypoeutectoid steel with a hole bored in it and the end closed, was allowed to cool through the critical range. On subsequent examination under the microscope, it was found that the hole was rimmed with ferrite. In order to ascertain whether or not this phenomenon was due to decarburization, a specimen of hypereutectoid steel was similarly prepared and heat treated in the same manner. In this case, examination showed the hole to be lined with cementite, showing that no decarburization had taken place.

Cold Work

Sheet steel is cold-rolled primarily to obtain good surface qualities, uniform gage, and flatness, i.e., freedom from buckles and waves. The reductions in thickness produced by cold rolling are seldom more than 3 per cent. Strip steel is cold-rolled for the same reasons, with the additional purpose of rolling to the lighter gages, particularly in the wider sizes. In the case of strip steel, the reductions effected by cold rolling are usually 20 per cent or more. If the sheet or strip is to be used for stampings in which the draw is only of moderate depth, a final pass on the cold rolls may be employed. This improves the surface and increases the "stiffness." To refer to one steel as being "stiffer" than another of the same thickness is incorrect because the rigidity (resistance to deformation) is the same in all steels. This is shown by the fact that the modulus of elasticity is practically a constant value: 30,000,000. A steel, therefore, which has been severely cold-rolled, when compared with one which has not been cold-rolled, will not resist a greater load before elastic deformation takes place, but the severely cold-rolled steel, having a higher elastic limit, can be deformed a greater amount and withstand a greater load before *permanent* deformation takes place.[11]

[10]Howe, "The Metallography of Steel and Cast Iron." p. 281. McGraw-Hill Book Co., Inc., N. Y.

[11]Moore, "Materials of Engineering." p. 38. McGraw-Hill Book Co., Inc., N. Y.

Mechanical deformation of iron and steel at low temperatures greatly affects the physical properties of these metals, the elastic limit, tensile strength, and hardness being increased, while the ductility, as represented both by the elongation and reduction of area, is decreased. The density of iron and steel is slightly decreased by cold working. The electrical resistance is slightly increased as is the solubility in acids and the tendency to rust or corrode. Evidences of the small reductions produced in cold rolling sheet steel cannot be detected in the shape of the grains by examination under the microscope.[12] Nevertheless, experience has shown that cold rolling as a final operation may markedly lower the deep drawing qualities of the steel.

The smaller the grain size prior to cold rolling, the greater is the work-hardening effect produced by a given amount of cold rolling. This appears, at first, to be inconsistent with the general observation that "A pass on the cold rolls will not lower the ductility of a small-grained sheet so much as it will a coarse-grained sheet." These statements are not contradictory, however, because the actual reduction obtained per pass on the cold rolls is greater in a large-grained material than that obtained in a smaller-grained material. Thus, "box-annealed" sheet steel, which generally has larger grains than strip steel or "normalized" sheet steel, is, when compared with these after the same number of passes on the cold rolls, the least ductile.

The percentage elongation in tensile specimens taken in a direction parallel to the direction of rolling is generally higher and the tensile strength and yield point generally lower than in specimens taken in a direction transverse to the direction of rolling. Cold rolling tends to accentuate this difference in that the rate of change to a lower ductility, for a given amount of reduction, is greater in the transverse direction than in the longitudinal direction. As a result of this, blanks for stampings are laid out, wherever possible, so that the maximum amount of the draw will take place in a direction parallel to the direction of rolling. And, in particular, bending of the metal flat on itself ("dutch bending") in a line parallel to the direction of rolling is avoided as much as possible.

A measurement of the increase in hardness by cold rolling and the decrease in hardness after subsequent annealing has not proved to be a reliable indication of the deep drawing qualities of steel because,

[12]Winlock and Kelley: "The Testing of Automobile Sheet Steel." TRANSACTIONS, American Society for Steel Treating. Oct. 1927. Vol. 12, p. 645.

as Jeffries and Archer point out: "A metal may be obtained with the same hardness by moderately working a coarse-grained piece or by annealing a severely worked piece at a temperature that will produce small unstrained grains. Although the hardnesses are the same, the other properties may not be, and usually are not, the same."[13]

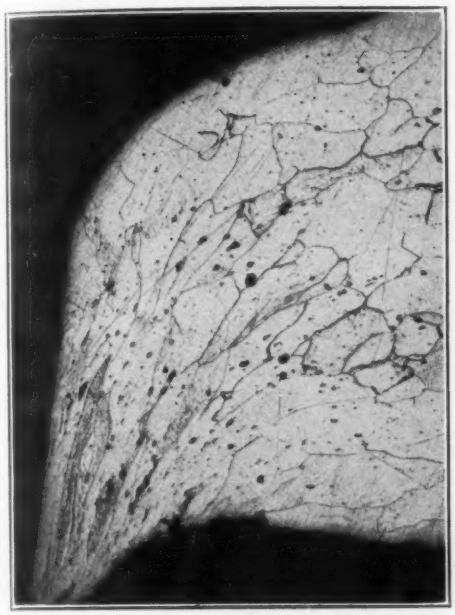


Fig. 8—Photomicrograph of a Sheared Edge of Sheet Steel Showing Elongated Grains Caused by Cold Work. × 75.

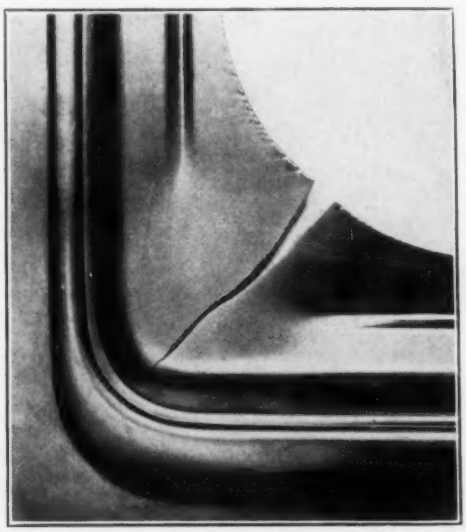


Fig. 9—Tears Resulting from Stretching a Blanked Edge.

In deep drawing, the elongation or stretching of a blanked or sheared edge is undesirable. This is because the blanking or shearing operation, as a result of the attendant cold work, causes a decrease in the ductility at the edge of the metal. Incipient tears may also be produced. The presence of these small tears in the edge of the steel causes, in drawing, concentrations of the imposed stress just as a small cut at the edge facilitates the tearing of a piece of coarse cloth. Fracture of the metal occurs, then, by a tearing action. Fig. 8 shows the microstructure of a sheared edge of sheet steel with elongated grains caused by the cold work, and Fig. 9 shows the tears which often occur as a result of the conditions just mentioned. The use of a blanking die which is not sharp or properly aligned obviously accentuates these conditions. Fractures due to these causes may often

[13]Jeffries and Archer, "The Science of Metals," p. 162. McGraw-Hill Book Co., Inc., N. Y.

be prevented either by filing or grinding away the damaged edge, or by annealing it.

If, after the yield point has been passed, the stress producing the deformation is removed, it will be found that instead of the stress having produced total permanent deformation, a slight amount of elasticity remains in the steel. This is shown in the stress-strain diagrams of the two steels in Fig. 10. It is evident that there are other forces existent in deep drawing operations in addition to those which are manifested in the stress-strain diagrams obtained in tensile testing. But owing to the fact that stress-strain diagrams which include all the forces with a given force predominating, are not at present available, it is believed that the stress-strain diagrams obtained in tensile testing lend themselves best to the discussion of the phenomena noted in deep drawing operations. As the stress is removed, the relation of stress to strain returns along the line CD for curve No. 1 and the line BA for curve No. 2, instead of along the perpendicular lines CE and BCE. The length of the lines DE and ADE is, then, a measure of the residual elasticity in the two steels respectively. In the deep drawing process, this phenomenon is known as "spring back." Upon further examination of Fig. 10, it will be seen that the greater the stress necessary to produce a given deformation, the larger will be the amount of spring back. The higher the tensile properties of the steel, therefore, the greater will be the amount of spring back for a given deformation. And, further, as the amount of the deformation increases, the amount of spring back will be larger the greater the rate of increase of the stress. Allowance must be made for this property of steel by so designing the dies for deep drawing operations that the metal will be deformed an amount greater than that desired in the finished stamping. It can be readily seen that if this were not done, the stamping would not be of the correct shape after removing it from the die. No general rules can be formulated regarding the proper allowances to be made for spring-back because the amount varies widely for each design of stamping.

It sometimes happens that upon applying a slight pressure of the hand to a stamping, a buckle will occur, and upon releasing the pressure, the metal will return to its original position. This phenomenon usually occurs in those stampings which contain a relatively large radius of curvature, such as automobile doors, tonneau-back panels, etc. It is due to the fact that the stamping has a low section modulus.

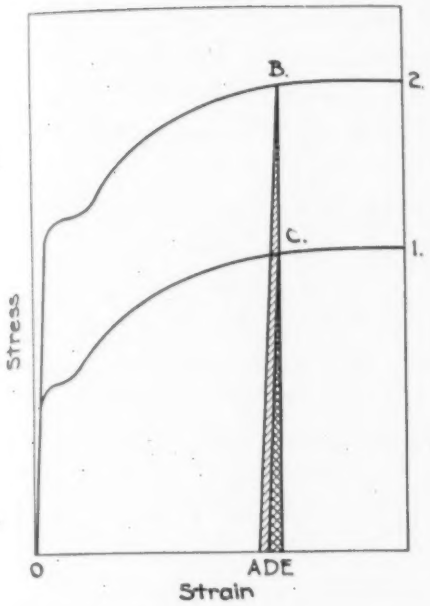


Fig. 10—Stress-Strain Diagram Illustrating "Spring Back."

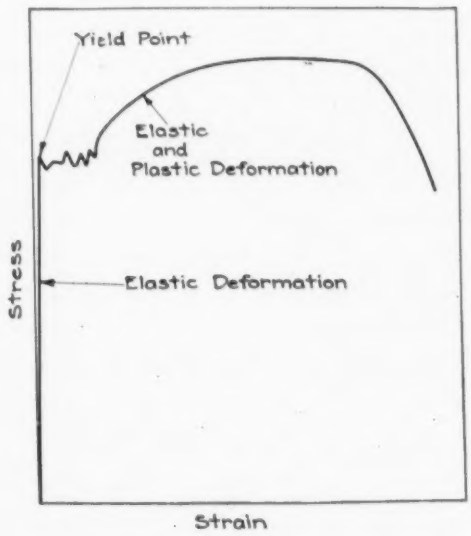


Fig. 12—Stress-Strain Diagram of Steel Specimen.



Fig. 11—Photograph of a Stamping Showing Stretcher Strains.

Low section modulus is due either to a poorly designed stamping, or to using a metal which is of too light a gage, or to not allowing for enough spring-back. In the latter case, the die may have been properly designed for a metal having a low elastic limit and tensile strength, but not for a metal having a high elastic limit and tensile strength. This results in a stamping having a slightly different shape (i.e., a larger radius of curvature) which has a lower section modulus.

A stamping should not, however, be considered defective if a buckle occurs in this manner, unless the stamping is securely fastened in the position in which it is to be used. This is because the constraining effect of the edges is often great enough to prevent the buckle from occurring.

The work necessary to deform steel may be expressed by the area under the stress-strain diagram (Fig. 12). This constitutes a measure of the "toughness" of the steel. For a given deformation, then, a greater amount of work will be required to deform a steel having a high yield point and tensile strength than is required to deform a steel in which these properties are lower. The average yield point of box-annealed sheet steel is approximately 25,000 pounds per square inch and the average tensile strength approximately 40,000 pounds per square inch. The average yield point of strip steel and "normalized" sheet steel is approximately 35,000 pounds per square inch and the average tensile strength approximately 50,000 pounds per square inch. Modifications in the design of the dies and changes in the adjustment of the presses are often necessary to compensate for such differences in properties as these.

As might be expected, straightening steel stampings by means of trip hammers lowers the ductility appreciably. This decrease in ductility is shown in the following tensile tests:

Longitudinal Tests (before hammering)		Transverse Tests (before hammering)
Elongation 2 inches	40.2%	29.7%
Elongation 8 inches	27.8%	22.6%
Yield Point	27,100	28,400
Ultimate Strength	43,700	44,700
Longitudinal Tests (after hammering)		**Transverse Tests (after hammering)**
Elongation 2 inches	28.3%	25.2%
Elongation 8 inches	20.5%	16.9%
Yield Point	35,700	44,700
Ultimate Strength	45,800	46,700

As a result of the hammering, the steel was reduced in thickness from 0.0415 inches to 0.0410 inches.

Stretcher Strains

When sheet and strip steels have been subjected to permanent deformation slightly beyond the elastic limit by stresses which are primarily tensile stresses, it sometimes happens that instead of the surfaces of the steel becoming uniformly roughened, irregular lines of depressions occur which destroy the evenness of the surfaces of the metal.* These markings are known as "stretcher strains" or,

*If the stresses producing the permanent deformation are primarily compressive stresses, irregular lines of *elevations* occur.

more appropriately, as "worms." In some cases, these depressions are so extensive and so deep that it is necessary to grind and polish the entire surface of the stamping. Even this is not always accomplished satisfactorily, because the heat produced results in a distortion of the stamping. Fig. 11 is a photograph of a stamping showing the appearance of stretcher strains.

Lüders,[14] Hartmann,[15] Cooper,[16] Breuil,[17] Gulliver,[18] Mason,[19] Turner and Jevons,[20] Rawdon,[21] and others have written accounts and published excellent photographs of their observations of this phenomenon met in the course of tensile testing.

The authors' conception of the modus operandi of stretcher strain formation and their observations regarding the prevention of this occurrence follows.

When a stress is applied to steel, the resulting deformation is either mainly elastic or mainly plastic according to the amount of the imposed stress. Elastic deformation is produced when the intensity of stress is of such a degree that no permanent deformation will remain when the stress is removed. In elastic deformation, the stress is proportional to the strain. This is shown in the stress-strain diagram in Fig. 12. If, however, the stress is of such a magnitude as to produce permanent deformation, the deformation is mainly of a plastic nature. The structure of the steel is changed: the crystalline grains being permanently elongated in the direction of the applied force.

The permanent deformation of steel takes place by a process of slip or gliding action within the crystalline grains. This slipping or

[14]Dinglers Polytechnisches Journal, 1860.

[15]"Sur la Distribution des Déformations dans les Métaux Soumis à des Efforts." *Comptes Rendus*, 1894, Vol. cxviii, p. 520.

[16]Bulletin of the Society of American Engineers, 1878.

[17]"Relations Between the Effects of Stresses Slowly Applied and of Stresses Suddenly Applied in the Case of Iron and Steel." *Journal*, Iron and Steel Institute, 1904, Vol. 64 (supplement).

[18]"Some Phenomena of Permanent Deformation in Metals." *Proceedings*, Institution of Mechanical Engineers, 1905. Parts 1 and 2.

[19]"Lüder's Lines on Mild Steel." *Proceedings*, Physical Society, 1910-11, Vol. xxiii, p. 305.

[20]"The Detection of Strain in Mild Steel." *Journal*, Iron and Steel Institute, 1925, No. 1, p. 169.

[21]"Strain Markings in Mild Steel under Tension." Bureau of Standards, *Journal of Research*, Sept., 1928.

gliding action does not take place in all of the grains simultaneously, but according to the resistance to slip of the different grains, or groups of grains. Resistance to slip is influenced principally by (1) the size of the grains, (2) the orientation of the grains in relation to each other, (3) the relation of this orientation to the direction of the stress producing the deformation, and (4) the slight concentra-

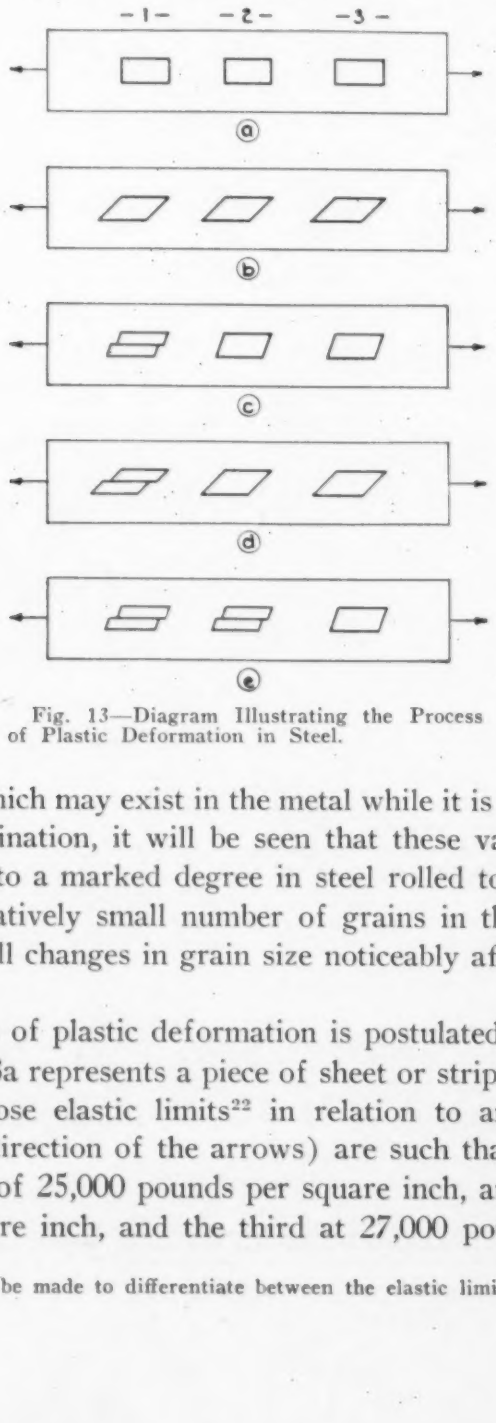


Fig. 13—Diagram Illustrating the Process of Plastic Deformation in Steel.

tions of stress which may exist in the metal while it is being deformed.

Upon examination, it will be seen that these various influences are accentuated to a marked degree in steel rolled to light gages because of the relatively small number of grains in the cross section, and because small changes in grain size noticeably affect the physical properties.

The process of plastic deformation is postulated as taking place in steps. Fig. 13a represents a piece of sheet or strip steel containing three grains whose elastic limits[22] in relation to an applied stress (shown by the direction of the arrows) are such that one grain will yield at a stress of 25,000 pounds per square inch, another at 26,000 pounds per square inch, and the third at 27,000 pounds per square

[22]No attempt will be made to differentiate between the elastic limit and the yield point.

inch. (The grains are shown as having the same orientation only for the sake of clearness.)

On application of a stress below 25,000 pounds per square inch, the resulting deformation will be elastic and the grains will assume the shape shown in Fig. 13 (b).[23] The material now conforms to the straight line denoting elastic deformation shown in the stress-strain diagram in Fig. 12. When a stress of 25,000 pounds per square inch is applied, the resistance to slip of grain number one is overcome, and a yielding of the grain takes place accompanied by the formation of a slip band. This produces a yielding of a group of grains which results in the formation of a stretcher strain. As a result of the yielding, the load momentarily drops off[24] causing a partial return of the two remaining grains to their original shapes. This is pictured in Fig. 13 (c). (The yield point of the metal as a whole, however, is reached at this point.)

Upon further application of the stress, the intensity of stress on the grain which has yielded is increased by virtue of the reduction in area, but due to work-hardening, the resistance to further slip is also increased. On application of further loads up to 26,000 pounds per square inch, the grains assume the shape shown in Fig. 13 (d). Upon reaching this stress, grain number two yields, bringing about in like manner the formation of another stretcher strain. This is shown in Fig. 13 (e).

As this alternate slipping and work-hardening continues, and the extent and number of the depressions in the surface of the metal increase, the effect of contrast continually diminishes until the condition is reached when the entire surface has been affected and the variations have become relatively minute. In effect, this is a return to the original even surface. When all of the grains have slipped in this manner, the stress-strain curve commences to rise again as a relatively smooth curve.

Stretcher strains are due, then, we believe, to the fluctuations occurring at the yield point caused by the comparatively sudden transitions in different grains, or groups of grains, from the elastic to

[23]The shearing stress is maximum at 45 degrees to the direction of the applied stress. (See Howe: "The Metallography of Steel and Cast Iron." p. 304.) Thus, the stresses on the faces of the crystals in relation to the direct forces are equivalent to a pair of equal and opposite couples.

[24]Robertson and Cook report that a reduction may occur in the load of approximately 27 per cent. ("The Transition from the Elastic to the Plastic State in Mild Steel." *Proceedings*, Royal Society, Vol. 88, 1913. p. 462.)

the semi-plastic state. In some cases, the fluctuations are so small that only a roughening of the surface of the metal takes place. Roughening of the surface and stretcher strains are manifestations of the same phenomenon. It is a question of degree and not kind. Their appearance is a normal characteristic of steel. A measure of the decreases and increases in load occurring after the yield point of the metal has been reached would, therefore, give an indication of the in-

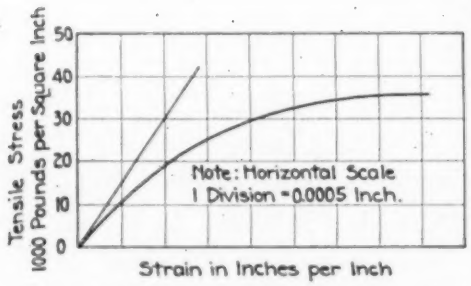


Fig. 14—Diagram Showing Effect of Overstrain on the Elastic Properties of Low Carbon Steel. (Van Den Broek).

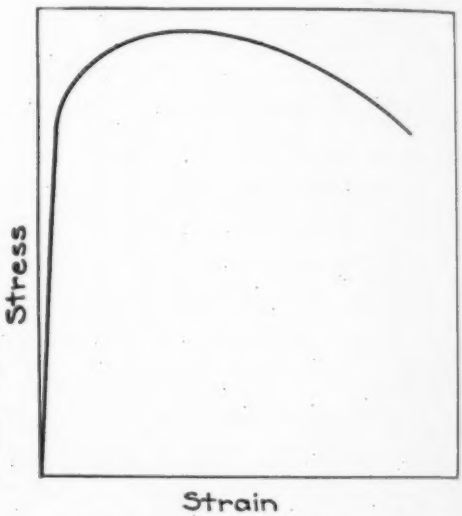


Fig. 15—Stress-Strain Diagram of a Steel Which Has Been Cold-Worked and Immediately Permanently Deformed. (Dalby).

tensity of the resulting stretcher strains. The elongations necessary to produce stretcher strains are between 1.5 and 10.0 per cent. They occur, then, in stampings or those parts of stampings where the amount of the draw is correspondingly slight.

Experiments have shown that the smaller the grain size of the metal, the greater will be the tendency for stretcher strains to appear. Where specimens with a small grain size showed deep depressions when the yield point was reached, specimens having only a few grains in their cross sections, in undergoing permanent deformation, showed only a roughening of the surface. It is interesting to note that the stress-strain curve of a steel having very large grains resembles closely those of the nonferrous metals which, as is well known, are characterized by a lack of definite yield point.[25] Experiments were also made on the nonferrous metals aluminum and copper in an attempt to pro-

[25]R. Arrowsmith, "A Note on the Effect of Grain Size on the Extension at the Yield Point in Armco Iron," *Journal*, Iron and Steel Institute, 1924, No. 2, p. 317.

duce stretcher strains. As was expected, however, only a roughening of the surface took place as in the case of the coarse-grained steel.

Let us now suppose that enough permanent deformation or cold work is imposed on the steel, before the deep drawing operation, so that all or most of the grains have partially slipped. Any subsequent deformation will be for the most part of a plastic nature. In Fig. 14 is shown a portion of the stress-strain diagram of a steel which has been cold-worked and immediately deformed, and in Fig. 15 is shown a complete stress-strain diagram of a steel which has been treated in the same manner. It will be noted that the stress-strain curve now departs from the straight line denoting elastic deformation, and the proportional limit has been reduced practically to zero. It can be seen also that the curve is smooth from origin to fracture. Stretcher strains will not occur when steel in this condition is permanently deformed.

Sheet and strip steel may be cold-worked to produce this condition either by cold rolling, roller leveling, or stretcher leveling. If the steel shows a tendency to kink or "break" in the roller leveling operation, this may be prevented sometimes by gradually increasing the amount of bending. Kinks are simply localized stretcher strains and should not be allowed to occur because the drawing operation may not always be of such an amount as to result in their removal. The amount of cold work necessary to prevent stretcher strains from occurring is relatively slight and in the case of roller leveling, even when using machines especially designed to produce drastic bending, a great number of passes have been found necessary to lower appreciably the ductility of the steel.

It is well known that steel in the "overstrained" state is in an unstable condition, and if the metal is permitted to rest or "age" or is heated slightly, it will recover its elasticity[26]. Muir[27] reports that elastic recovery at 210 degrees Fahr. (100 degrees Cent.) is as complete after a few minutes as in two weeks at room temperature, and is impeded or entirely prevented at lower temperatures, i.e., around 32 degrees Fahr. On recovery, the steel will have a new elastic limit higher than that of the original material and will be slightly harder.

[26]Stromeyer, "The Aging of Mild Steel," *Journal*, Iron and Steel Institute, No. 1, 1907, p. 200; 1907, No. 3, p. 86.
McCaustland: "Effect of Temperature on the Recovery of Steel from Overstrain." *Journal*, Iron and Steel Institute, No. 4, 1906, p. 616.

[27]"On the Overstraining of Iron," *Philosophical Transactions*, Royal Society of London. A 193, p. 1; A 198, p. 1, *Proceedings* A 77, p. 277.

The experience of the authors has shown that for consistent results, the steel should be roller leveled within the twenty-four hour period preceding the drawing operation. Cold rolling is a more drastic form of cold work and consequently longer intervals may elapse before the metal will recover from the effects of the overstrain thus produced.

Obviously, then, if a local annealing operation is necessary between two deep drawing operations, precautions must be taken to prevent the whole stamping from being heated, otherwise stretcher strains may occur in the second operation.

As a result of the plastic behavior of steel in the overstrained state, the apparent "strength" of cold-worked steel is decreased on



Fig. 16—Sketch Showing the Effect of Local Deformation and Aging of Sheet Steel After Drawing.

account of the ease with which a permanent "set" may be produced. In the experience of the authors, it has been found desirable, in some cases, to heat the steel slightly after forming in order to hasten the recovery of its elastic properties. An example showing the necessity of such a treatment was in the manufacture of locking rings. These are split rings sometimes used to lock automobile tires to the rims. After the steel had been rolled into the desired circular shape, it was found that the elasticity necessary for the proper functioning of the ring was lacking. Only a slight force tending to spread the ends of the ring was necessary to produce a permanent distortion of the ring. Heating it, however, for a short time at about 300 degrees Fahr. (150 degrees Cent.) completely restored the elasticity.

There is another phase of the phenomenon which is worthy of mention. As has already been pointed out, the irregular lines of depressions "disappear" when the steel is elongated well beyond the yield point because the effect of contrast diminishes as the stretch increases until the entire surface has been affected. If, however, any previous local deformation has occurred, such as might be produced by kinking in cold rolling or roller leveling, etc., subsequent drawing, provided, of course, that aging of the metal has taken place, will not remove the traces of the local deformation even though the yield point is greatly exceeded. In this case, the locally deformed areas will stand in relief, after drawing, as ridges or slightly raised veins on account

of the fact that they do not elongate as much as the surrounding metal. In Fig. 16 is a sketch showing this effect. The shaded area represents the thickness of the sheet prior to forming. At A is a local deformation caused by a stretcher strain, and at B a kink which may not have produced any visible deformation. The solid black portion of the sketch indicates the thickness of the sheet after it has been drawn showing the ridges and veins formed in this manner. This may be easily demonstrated experimentally by pulling in a tensile machine a specimen of sheet or strip steel which has been heated to about 1200 degrees Fahr. (650 degrees Cent.) and cooled in air to remove any existing cold working strains. When the depressions begin to form, the specimen is removed from the machine and warmed slightly for a short time. Repulling the specimen to fracture will not cause the original depressions to "widen" and disappear as usual, but will, in effect, now cause them to stand in relief. During the drawing of a steel in this condition, a new set of stretcher strains may appear, and disappear when the metal has been elongated well beyond the yield point, but the occurrence of these may be prevented by roller leveling.

Sheet or strip steel may be prevented from exhibiting the effect of such local deformations and aging by annealing at approximately 1100 degrees Fahr. (590 degrees Cent.).

Annealing

The "box-annealing" process and the "normalizing" process are the two principal methods used for annealing sheet and strip steel. As the name implies, the former is carried out in boxes inserted in furnaces. The temperatures employed in box-annealing are below the Ac_3 point of the steel [usually between 1150-1400 degrees Fahr. (620-760 degrees Cent.)]. The normalizing process is carried out in continuous type furnaces above the Ac_3 point [about 1700 degrees Fahr. (925 degrees Cent.)]. In box-annealing, the sheet metal is annealed in piles, whereas in the normalizing process each piece is annealed individually. The maximum temperature obtainable in the box-annealing of sheet steel and strip steel cut to the shorter length is governed by the quality of the surface and the weight of the charge. In general, the better the surface qualities, the lower is the temperature which can be reached because of the greater tendency toward welding ("sticking"). This tendency is increased as the weight of

the charge increases. Strip steel, when annealed in coils placed in upright positions in boxes, is not subject to this difficulty to any appreciable extent, with the result that higher temperatures may be used.

In the authors' opinion, the deep drawing qualities of sheet and strip steel are dependent not only upon the physical properties of the metal, but also upon the magnitude and distribution of the forces producing the specific deformation.[28] For example, let us assume that the two different shapes shown in Fig. 17b and 17c are to be

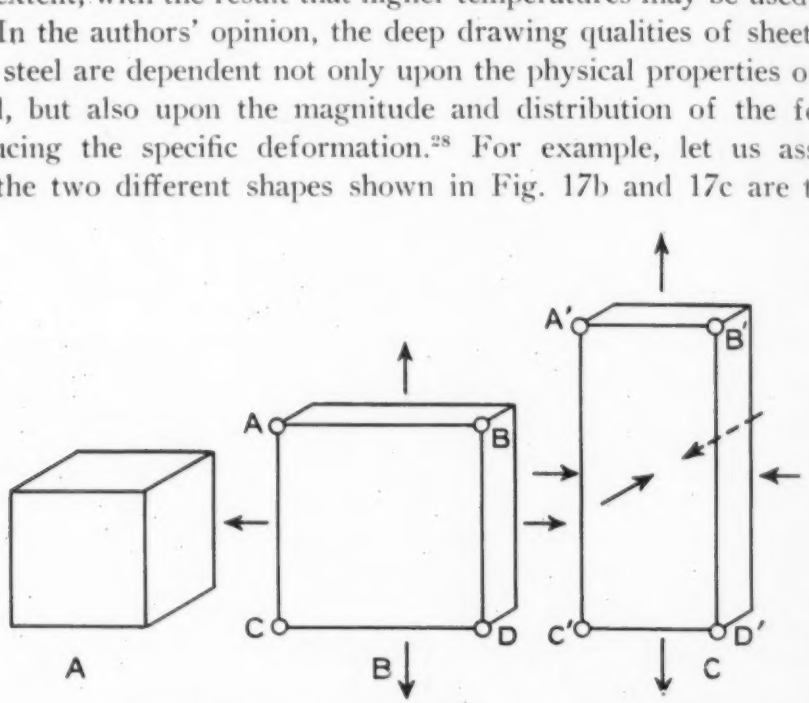


Fig. 17—Diagram Showing Some Effects of a Different Distribution of Forces in Deep Drawing.

formed by separate drawing operations, from the shape shown in Fig. 17a, so that the resultant areas A B C D and A^1 B^1 C^1 D^1 will be the same. And, further, let us suppose that the forces which are to produce the deformation will act in the direction designated by the arrows. Experience indicates that the physical properties of a steel which will satisfactorily form 17b are different from those of a steel which will satisfactorily form 17c.

In studying the relation between the different types of deformation produced by deep drawing, the experience of the authors has been that, in the annealed condition, the adaptability of the steel for deep drawing is influenced by the size of the grains more than by any other factor. Further, that there is no difference in the deep drawing qualities of sheet steel or strip steel, provided the grain size is the same, and that within certain limits, a small rather than a large grain size is more

[28]For a discussion of the different forces acting on the metal in deep drawing, see Eksergian: "The Plastic Behavior of Metal in Deep Drawing." *Transactions*, American Society Mechanical Engineers, Dec. 1926.

adaptable for most deep drawing operations. Inasmuch as only a few experiments have been completed showing the effect of small differences in grain size, only a few general observations can be given.

There are two conditions to be avoided in annealing steel for deep drawing purposes: the first is the production of too large a grain size, and the second is the production of too small a grain size.

A sheet metal having a large grain size will exhibit, after deep drawing, a coarse granulated surface. This requires filing and polishing operations in order to obtain a smooth surface on the finished stamping. The degree of coarseness is directly proportional to the grain size. In Fig. 19 is shown the microstructure of a specimen of sheet steel which will produce this coarseness.

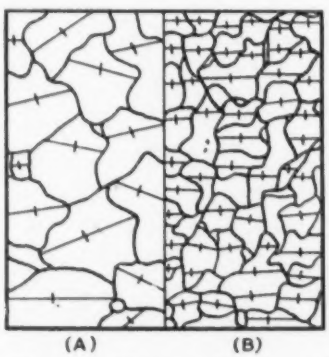
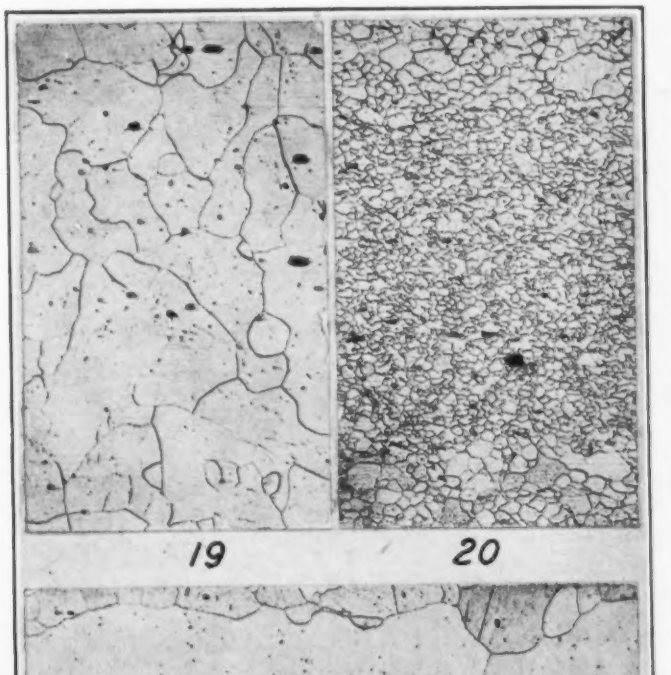


Fig. 18—Diagram Showing (A) Cleavage Planes Not in a Straight Line. (B) Cleavage Planes in a Straight Line.

A steel having a large grain size is subject to the phenomenon known as "Stead's brittleness" more often than a steel of small or medium grain size. This is a peculiar type of brittleness which sometimes occurs in low carbon steel.[29] It occurs on annealing after cold working and is due to the crystalline grains assuming the same or nearly the same orientation.[30] The cleavage planes are almost in a straight line, as is shown in Fig. 18. Steel in this condition is very susceptible to fracture by dynamic stresses. Cases have been cited by Stead where fracture has occurred by simply dropping the metal on the floor. Fractures resulting from this type of brittleness usually occur when the major stress is at 45 degrees to the direction of rolling. Thus a steel may draw perfectly in a preliminary operation and then

[29]Stead: "Brittleness Produced in Soft Steel by Annealing." *Journal,* Iron and Steel Institute, No. 2, 1898, p. 137.

[30]Jeffries and Archer, "Science of Metals," McGraw-Hill Book Co., p. 206.



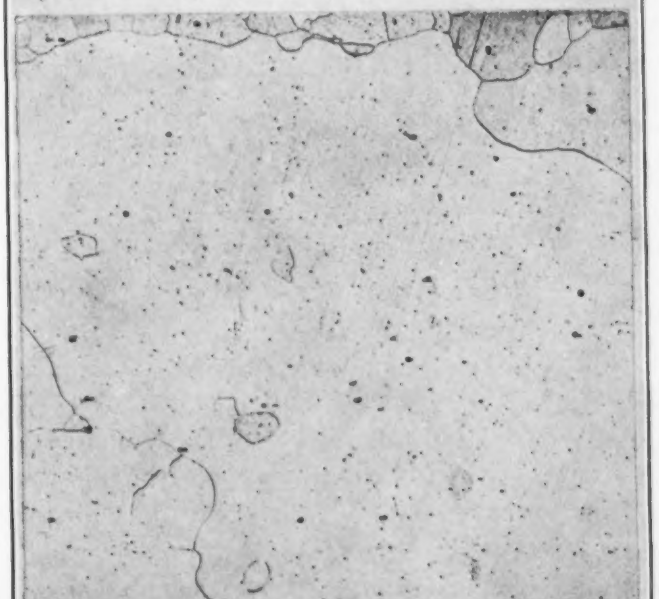


Fig. 19—Photomicrograph of Specimen of Sheet Steel Showing Coarse Grains.
Fig. 20—Cross Section of a Specimen of Sheet Steel Showing Very Small Grains.
Fig. 21—Cross Section of a Specimen of Sheet Steel Showing Exaggerated Grain Growth. All Photomicrographs × 75.

exhibit extreme brittleness in a subsequent operation in which the application of the stress has been from a slightly different direction. Straightening or flattening stampings by means of trip hammers often causes fracture of steel in this condition. Fig. 22 is a photograph of



Fig. 22—Photograph of a Portion of a Stamping Showing Profile of Fracture Peculiar to Stead's Brittleness.

a portion of a stamping made from a steel which exhibited this type of brittleness and shows the profile of the fractures obtained. (That a steel having large grains is not always brittle is shown in Fig. 18 A.)

Sheet metal having too small a grain size, such as is shown in Fig. 20, exhibits in too great a degree the property already referred to as "toughness." On account of its high yield point and tensile strength, the work necessary to form a steel of this kind is sometimes so great as to make its use prohibitive by the ordinary methods of deep drawing. Steel having a very small grain size frequently causes the formation of buckles in the stamping, and, in some cases, causes excessive wear on the dies.

When sheet steel, after being cold-rolled, is heated to the temperature zone indicated by the heavy black line in Fig. 2, a recrystalliza-

tion of the metal takes place. This results in the removal of the strains produced by the cold work. The temperature at which recrystallization is completed, the size of the grains obtained when recrystallization is complete, and the size of the grains resulting from subsequent growth on further heating to a higher temperature or for a longer time at the same temperature, depend upon certain conditions existing in the steel.

In addition to the recrystallization taking place as the result of heating after cold work, another recrystallization of the metal occurs if the metal is heated above the Ac_3 point. This recrystallization takes place in the normalizing process. The size of the grains resulting from this type of annealing are generally small on account of the comparatively rapid rate of heating through the critical range, the short time held at the maximum temperature, and the comparatively rapid rate of cooling from the high temperatures reached. By governing the rate of heating, the time at temperature, and the rate of cooling, the size of the grains may be accurately controlled. With a proper arrangement of these rates a "soft" sheet (i.e., having low elastic limit and tensile strength) may be obtained without necessarily producing a grain size too coarse to be consistent with good practice.

The phenomena of recrystallization on annealing after cold work, and grain growth in iron and steel have been extensively described by many investigators. Stead,[31] Howe,[32] Sauveur,[33] Chappell,[34] Jeffries,[35] Edwards and Pfeil,[36] and many others have published very valuable papers on this subject. In the papers by Jeffries discussions of the laws governing grain growth are given. Those laws which apply particularly to the grain size changes occurring in sheet and strip steel during annealing, follow:

(1) The recrystallization temperature is lower: (a) the greater

[31] "The Crystalline Structure of Iron and Steel." *Journal,* Iron and Steel Institute, 1898, No. 1, p. 145.

[32] "Recrystallization after Plastic Deformation." *Transactions,* American Institute of Mining and Metallurgical Engineers, Vol. 56, p. 561.

[33] "Note on the Crystalline Growth of Ferrite below its Critical Range." *Proceedings,* International Association for Testing Materials, Sixth Congress, New York, 1912, Vol. ii.

[34] "Recrystallization of Deformed Iron." *Journal,* Iron and Steel Institute, 1914, No. 1, p. 460.

[35] "Grain Growth and Recrystallization in Metals." *Chemical and Metallurgical Engineering,* 1922, Vol. 26, p. 449. "Grain Growth in Metals." *Journal,* Institute of Metals, 1918, No. 2, p. 109. "Grain Growth and Grain Growth Phenomena in Metals." *Transactions,* American Institute of Mining and Metallurgical Engineers, Vol. 56, p. 571, 1916.

[36] "The Production of Large Crystals by Annealing Strained Iron." *Journal,* Iron and Steel Institute, 1924, No. 1, p. 129.

the amount of deformation, (b) the smaller the grains prior to deformation, (c) the purer the metal, (d) the lower the temperature at which deformation is effected, and (e) the longer the time of heating. (2) In any given metal, the grain size after complete recrystallization is normally smaller, the lower the recrystallization temperature. (3) Above the recrystallization temperature, the grain size is normally greater, the higher the temperature and the longer the exposure.

In summarizing the effects of annealing cold-rolled steel, then, it may be seen that:

(1) The size of the grains in strip steel and normalized sheet steel are generally smaller than in box-annealed sheet steel.

(2) If the temperature reached in box-annealing is too high, a coarse grain may result.

(3) If the time at temperature in box-annealing is too long, a coarse grain may result.

(4) If the time at temperature in normalizing is too long or the rate of cooling too slow, a coarse grain may result.

(5) If the temperature reached in box-annealing strip steel which has been cold-rolled severely is too low, and if the time at this temperature is too short, the size of the grains will be very small.

(6) If the rate of cooling from the normalizing temperature is too fast, the size of the grains will be very small. (The effect of this increases as the carbon content of the steel increases.)

(7) A small uniform grain size may be produced by box-annealing strip steel or by normalizing either strip steel or sheet steel.

(8) If the surfaces of the metal have been decarburized, large surface grains may result.

Sometimes, in annealing low carbon steel, the phenomenon known as "exaggerated" or "critical" grain growth occurs. This was first investigated by Sauveur who found that a low annealing temperature, 1200 degrees Fahr. (650 degrees Cent.), caused the development of a coarse crystalline structure in low carbon steel which had been subjected to a certain amount of cold plastic deformation. Where the strain was more or less than this amount, heating to this temperature did not produce a coarse grain. It occurs only when the carbon content of the steel is below some 0.15 per cent.

The condition which most readily results in the occurrence of

exaggerated grain growth is a reduction in cold rolling of about 5-15 per cent followed by heating at various temperatures from 1150 degrees Fahr. (620 degrees Cent.) to the Ac_3 point of the steel. Exaggerated grain growth is also dependent upon time for its development. Slow heating to temperature and sufficient time at temperature are, therefore, also necessary. Exaggerated grain growth seldom occurs in strip steel on account of the much larger reductions used in cold

Fig. 23—Graphs Showing Decrease in Elongation of Boiler Plate Steel at Elevated Temperatures.

rolling. Exaggerated grain growth may sometimes occur in sheet steel, which has been box-annealed at these temperatures, as a result of "critically" straining the steel by finishing at too low a temperature on the hot mill, by an unusual amount of cold rolling, or by the combined effect of a small amount of cold work on the hot mill followed by the usual amount of cold rolling. The presence of temperature gradients, strain gradients, and grain size contrast also favor large grain growth.

While it is true that exaggerated grain growth is subject to the same laws as those which govern normal grain growth, it presents certain peculiarities in that the degree of coarseness of grain is often greater than could otherwise be produced except by the use of extreme temperatures or very long periods of heating. And, further, it is subject to certain perverse irregularities in its appearance. Fig. 23 shows the microstructure of a specimen of sheet metal showing the very large grains resulting from this phenomenon.

Large grains produced in sheet steel or strip steel, whether or

not they have been produced in this manner, can be removed by either severe cold rolling followed by annealing at low temperatures or by heating the metal above the Ac_3 point. Exaggerated grain growth may sometimes be prevented from occurring by causing a partial removal of the strain by a preliminary heating below the recrystallization temperature of the metal.[37]

A reducing atmosphere is often produced in box-annealing by introducing carbon monoxide or illuminating gas into the boxes during heating and cooling. When this is done, discoloration of the sheets or strip is prevented which would otherwise occur as a result of the formation of slight films of oxide. In the experience of the authors, neither the presence nor the absence of this discoloration in any way affects the physical properties of the steel for deep drawing purposes.

Stampings which have been annealed so that a subsequent drawing operation can be performed must be allowed to cool thoroughly to atmospheric temperature, otherwise the steel will show a tendency to fracture. The decrease in ductility of steel, as expressed by the percentage elongation, at slightly elevated temperatures is shown[38] in Fig. 23.

DISCUSSION

DR. W. R. FRAZER[1]: This splendid paper by Mr. Winlock and Dr. Kelley certainly shows the thoroughness with which these gentlemen have studied the problems connected with their particular industry. I have just been wondering if any of the phenomena of "blue brittleness" are encountered in deep drawing operations.

A. L. DAVIS[2]: I should like to ask Mr. Winlock what he considers a large grain size; and what he considers a normal one?

We have considered a normal grain size in strip steel as anywhere between 0.02 and 0.05 mm. When it increases beyond these figures we consider the size large. We have seen it as large as 0.09.

E. S. LAWRENCE[3]: I have three questions which I should like to ask Mr. Winlock. (1) In the first part of the paper he mentioned chemical analysis. He said that the carbon content was lower than 0.14 per cent and that the

[37]Kelley and Winlock, "On the Restraint of Exaggerated Grain Growth in Critically Strained Metal." *Journal*, Franklin Institute, Jan. 1926, p. 71.

[38]French, "Strength and Elasticity of Boiler Plate at Elevated Temperatures." *Chemical and Metallurgical Engineering*, Vol. 26, p. 1207.

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[2]Member A. S. S. T., Metallurgist, Scovill Mfg. Co., Waterbury, Conn.

[3]Member A. S. S. T., Metallurgist, Duraloy Co., Pittsburgh.

manganese was often as low as 0.30 per cent. I should like to ask if he knows the effect of small changes in the manganese content. (2) I should like to ask what would be the physical properties of the steel which would be best suited for fender stock, particularly as regards "stiffness." (3) In speaking of stretcher strains, Mr. Winlock spoke of the fluctuations occurring at the yield point. I should like to ask if operating the presses at variable speeds, thereby producing various rates of deformation would have any effect on the occurrence of stretcher strains.

G. H. BIERMAN[4]: I should like to ask Mr. Winlock if the so-called "rimmed" steel which has a large number of blow holes in the ingot but which weld up in the rolling operations is used in the manufacture of sheet and strip steel.

E. S. TAYLERSON[5]: It is seldom that the authors of a technical paper are so familiar with both the practical and theoretical sides of their subject. I think that this paper will, in the future, be regarded as a classic on the subject of the deep drawing of low carbon sheet steel. From the amount of work which the authors have presented, it is apparent that the problem is a difficult one. Their clear conclusions on the nature of the intermittent deformation of low carbon steel are confirmed by the work of Turner and Jevons[6] referred to in the paper and by other investigators using the Fry etching reagent. The extension of a tensile specimen is found to occur first at the fillets in definite wedge-shaped bands which gradually extend to the middle of the gage length. These irregular local elongations are identical with the "stretcher strains" which the authors have so ably described in this paper.

They have also brought out in a very clear manner the fact that there are two distinct types of annealing:

(1) Annealing for grain refinement and the relief of hot rolling stresses,
(2) Low temperature annealing which does not change the microstructure, but relieves the internal stresses due to cold work.

As this is the first time that this subject has been so clearly discussed, it is my belief that this paper will be of great use not only to the deep drawing and automobile industries, but also to the manufacturer of sheet steel.

JOSEPH WINLOCK: I believe that the phenomena connected with "blue brittleness" are ever present. It is probably because we do not know a great deal about them that they are so peculiarly elusive. They hang over our heads like the sword of Damocles. But we prefer to recognize them as something to overcome by further research rather than to place our trust in the proverbial hair.

There are several theories relating to the cause of "blue brittleness."

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[5]Member A. S. S. T., Engineer of Tests, American Sheet and Tinplate Corp., Pittsburgh.

[6]*Journal*, Iron and Steel Institute, Vol. CXI, 1925, p. 169 and 191.

Pfeil[7] believes that the precipitation of cementite from solid solution in alpha iron is the cause. Dean, Day and Gregg[8] attribute it to a solution of precipitated nitrides. Balsover[9] believes that it may be due to the formation of submicroscopic crystals at the grain boundaries. Archer[10] believes that the blue heat properties are due to a certain peculiarity of the process deformation, i. e., an increase in resistance to motion on the slip planes which increases as the temperature is raised. Grossmann and Snyder[11] explain the phenomena along somewhat similar lines. I think it is a fairly well established fact that the phenomenon occurring at 480 to 750 degrees Fahr. (250 to 400 degrees Cent.) and the slow changes in properties which take place at room temperature after cold working ("ageing") and the changes in properties which occur on heating cold-worked steel to these temperatures are due to the same cause. Stretcher strains appear in material which is in the elastic condition or which after being cold-worked is put into the elastic condition. In cold-worked materials which have been so conditioned at low temperatures that stretcher strains will appear, blue brittleness may appear also. Brittleness is sometimes encountered in cold-worked low carbon steel after welding or local annealing. It occurs in those portions of the metal adjacent to the local heating. The susceptibility of the metal to fracture when deformed at slightly elevated temperatures is also connected with these phenomena.

We have no exact figures, as yet, for the normal grain size from the point of view of the capacity of the steel for deep drawing purposes or figures for a grain size which would indicate the degree of coarseness of the surface after drawing.

We do not know the effect of small changes in manganese content. We are led to believe from our experiences with some steels containing 0.04 per cent manganese and some 0.15 per cent carbon which gave excellent results that, with everything else being equal, the lower the manganese, the more ductile the steel.

In most cases we do not rely on the inherent physical properties of the steel for rigidity in the finished stamping. Rigidity is imparted to the stamping by designing it so that it has a high section modulus. For example, we can increase the range of elastic deformation in a fender of a given design by increasing the elastic limit or "stiffness" of the steel but the rigidity of the fender cannot be increased except by a change in design or by using steel of a heavier gage.

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[7]L. B. Pfeil: "The Change in Tensile Strength due to ageing of Cold Drawn Iron and Steel." *Journal,* Iron and Steel Institute No. 2, 1928, p. 167.

[8]R. S. Dean, R. O. Day and J. L. Gregg: "Relation of Nitrogen to Blue Heat Phenomena in Iron and Dispersion Hardening in the System Iron-Nitrogen." *Transactions,* American Institute of Mining and Metallurgical Engineers, February, 1929.

[9]G. R. Balsover: "Brittleness in Mild Steel." *Journal,* Iron and Steel Institute, No. 1, 1929, p. 473.

[10]Discussion of paper by Zay Jeffries: "Physical Changes in Iron and Steel Below the Thermal Critical Range." *Transactions,* American Institute of Mining and Metallurgical Engineers. Vol. LXVII, 1922, p. 73.

[11]M. A. Grossmann and C. C. Snyder: "Hardening by Reheating after Cold Working." TRANSACTIONS, American Society for Steel Treating, Vol. 13, Jan. 1928, p. 201.

In our experience, within the limits of the speed of our presses and those obtained in an ordinary tensile testing machine, the rate of deformation does not affect the occurrence of stretcher strains.

Both "rimmed" or "effervescent" and "killed" steels are used in the manufacture of sheet steel and strip steel for deep drawing purposes. The present tendency is, however, to use the "rimmed" steel almost entirely. If the "rimming" process is correctly carried out, an ingot is produced which has a thick skin of dense metal surrounding a core of spongy metal which contains most of the impurities. The blow holes in the center of the ingot readily weld up in the subsequent rolling processes and the thick skin of metal results in better surface qualities.

I wish to thank Mr. Taylerson for his kind remarks. He emphasizes in his discussion the fact that there are, in general, two types of annealing: low temperature annealing (below Ac_3) and high temperature annealing (above Ac_3). The mechanism of the recrystallization which takes place at low temperatures is, of course, somewhat different from that which takes place as the steel passes through the Ac_3 point. In the former, we are dealing with an aggregate of alpha iron (ferrite) and pearlite or spheroidized cementite. In this case, recrystallization (if it really may be called this, since there is no change in atomic arrangement) takes place by a growth of the small grain fragments produced by the cold work. In high temperature annealing, we are dealing with a more or less homogeneous solid solution of carbon in gamma iron (austenite). In both cases, the size of the resultant grains is governed by the usual laws of grain growth in which time, temperature, temperature gradients, grain size, grain size contrast, etc. take such important parts. In low temperature annealing, there is the additional influence of strain and strain gradients. It should also be emphasized, then, that the effect of the *amount* of reduction by cold rolling is greater in low temperature annealing than in high temperature annealing. When the reductions are small as is the general practice in the manufacture of sheet steel, there is usually no marked change in the size of the grains as a result of a low temperature anneal. In strip steel, however, the reductions by cold rolling are usually 20 per cent or more. In this case, we may obtain, and usually do, a decrease in the size of the grains as a result of the large reduction in cold rolling and the low temperature anneal. In effect, an actual refinement of the grains relative to the original grain size is produced.

THE PHYSICAL PROPERTIES OF FINE BOLTS

By H. B. Pulsifer

Abstract

The nominal properties that may be expected in bolts made from the usual materials are determined by from one to five hundred tests on each material. The question of uniformity and spread in properties is primarily studied by using single coils of wire and single batches in heat treatment.

The results are assembled in eleven charts, each of which has the distribution curves for the yield point, tensile strength, hardness and elongation of one material. Over 3000 bolts were tested for these curves. These graphs are a provisional estimate of the properties and precision that may be obtained with current steels and present manufacturing methods. Photomicrographs show typical structures of the materials as tested. The strengthening effect of threads is measured by a series of special tests comparing the physical properties of the normal bolts with test bolts machined to the root diameter of the threads. The yield point is usually increased more than the tensile strength and the amount ranges from six to twenty-six per cent.

IT is well known that the statistical method, as worked into probability curves and formulas, is highly useful in many fields of human interest. The method is classic in the insurance business, gambling propositions, and biological science. The method affords exhilarating exercises for the mathematician and is important in the science of measurements. Various engineering problems are now being clarified by the method and a very significant report by Janitzky[1] relates to the properties that may be expected of a chromium-vanadium steel.

Anticipating that study of the properties of bolts made from various steels may be of some utility, the following data have been

[1] E. J. Janitzky, "Correlating Test-Data on Heat-Treated Chromium-Vanadium Steels." S.A.E. *Journal*, Vol. XXII, 1928, p. 55.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, H. B. Pulsifer, member of the society, is metallurgist for the Ferry Cap and Set Screw Company, Cleveland, Ohio. Manuscript received April 30, 1930.

accumulated on the subject. It is evident that this paper can be no more than a preliminary outline, for the very statement of the data suggests further and more complete investigation that should be undertaken. It is also suspected that great quantities of similar data are on hand in laboratory records and that some of it may venture out after the limitations of this present study are apparent.

The reasons for the statistical method in studying the properties of bolts are obvious but should be stated for the benefit of those not in close contact with the industry.

Practically all bolts and cap screws of the finer grades are bought and sold under some physical specification. The requirement is usually that all the pieces be within some range of Brinell hardness. About 50 units is a common "spread." Brinell 207 to 255 is a common range for treated bolts of straight carbon steels containing 0.30 to 0.40 per cent carbon. Brinell 269 to 321 is a common range for treated bolts containing nickel and of about the same carbon range. But other requirements may be some minimum yield point, or minimum tensile strength. And that no excessive hardness or brittleness should be present it is customary to add a bend test, an impact test or even a hardness range on either Scleroscope, Rockwell or Brinell scales.

Buyer and seller are accordingly very much concerned about the properties of bolts and especially the "distribution" or spread of tests that is inevitable in commercial products. Since all probability curves have most frequent values, adjacent values occurring less frequently, and extreme values that occur rarely, we may expect this condition to be found in our tests on bolts. Our bolt studies will probably appear extremely elementary to the mathematician, even crudely so, because of the relatively few tests it is practicable to make. One notices that where smooth and symmetrical curves are described in the literature it is not uncommon to have some thousands of tests available for a single curve. The facilities available have allowed a maximum of only 500 tests for single curves and usually the number is less. The small number of tests will tend to make the curves irregular. But that does not matter for we are not seeking formulas for the curves, merely plotting values against the frequency of their occurrence.

The graphs will give some idea of the mean values for the various materials and treatments, the average deviations, the width of the ranges, and some sort of an idea as to how many pieces per

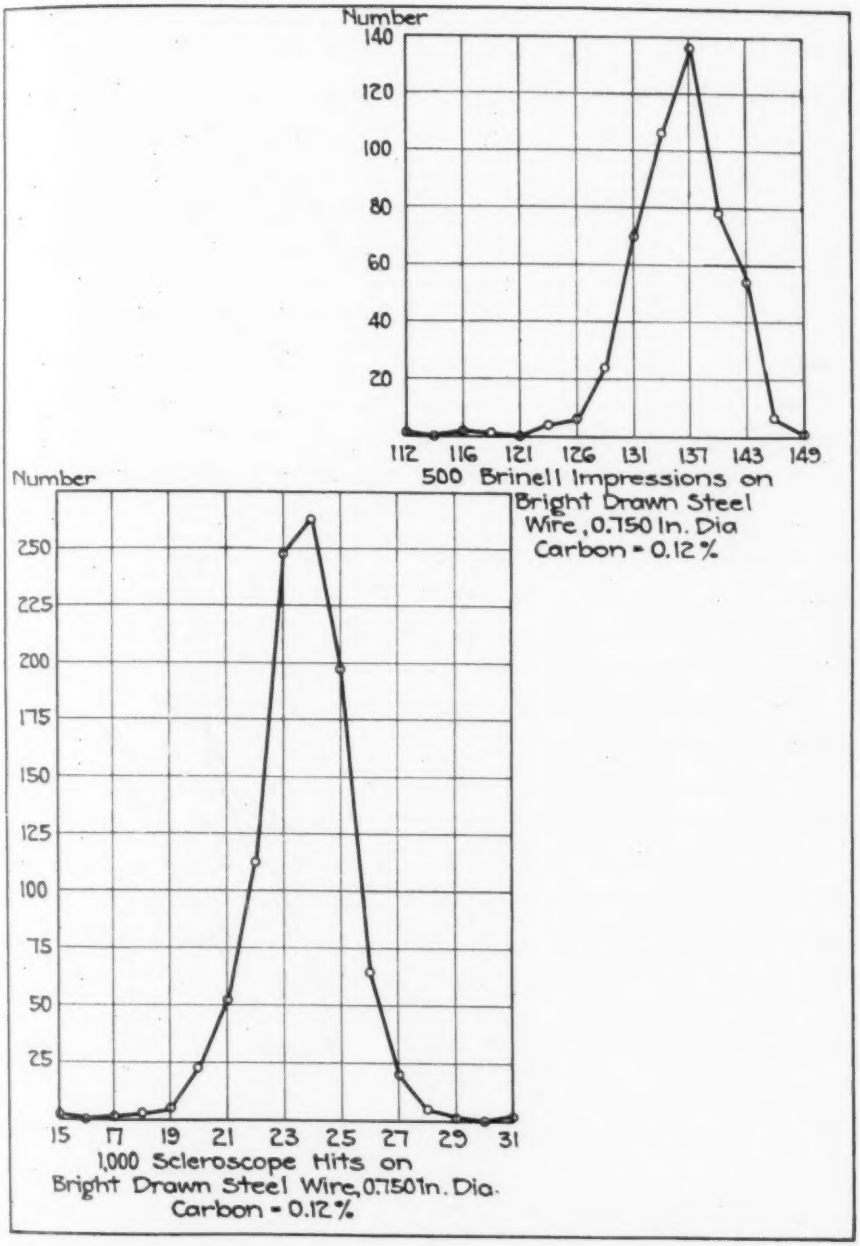


Fig. 1—Brinell and Scleroscope Tests on Bright Drawn Steel Wire.

hundred may be expected to fall outside of given ranges.

The heat treated lots were heated for quenching in gas-fired retort furnaces of the rotary type. From the retorts the bolts were raked out to slide down a short trough into the oil quench. The quenching tanks were of the well-known Greene type, the oil being

circulated against the bolts with a pump and the bolts caught and elevated in a rotating perforated spiral drum. The bolts were drawn in electric furnaces of the salt pot or Homo type. The tensile testing machine pulled at the rate of 0.05 inch per minute with a load of 10,000 pounds.

With exception of the cap screws tested in the chart on "milled-from-bar" screws, all of the bolts were made by the cold up-set method. All of the bolts tested failed in the threaded section. No heads came off. The matter of thread size and root diameter may be important and might give independent distribution curves if the facts could be segregated. In this study, however, it is held that the root diameters have not influenced the results. The conditions of manufacture were such that very small variations occur in pitch or root diameter. Measured pitch diameters agree closely and usually less than 0.002 inch below the base figure. One lot of 150 bolts, not here recorded, was measured and separated into two lots as passing a certain micrometer setting either snugly or loosely. There was no correlation with tensile results and the average of the two lots was practically identical. All of the results in this paper are figured on the root diameters.

The question of discarding "freak" results is highly important. In the report by Janitzky there was a discard of the most erratic results. In the tests here recorded there were no discards. For the purpose of discovering the facts of uniformity and spread in properties, one of the chief aims of these investigations, the author maintains that it would be grossly in error to discard any results however discordant.

Results of Tests

The results recorded in Fig. 1 are of an introductory nature to indicate the distribution of results as found by testing a single length of wire that might be considered as "uniform."

A section from a coil of 0.750 inch wire was cut into convenient lengths, care being taken to avoid surface injuries. The microscopic structure of the wire is indicated in longitudinal section by Fig. 14 which is at 200 diameters magnification.

A thousand scleroscope hits were made in spirals around the wire. The most frequent result is 24. The curve is nearly symmetrical with many results near the most frequent and a few wide

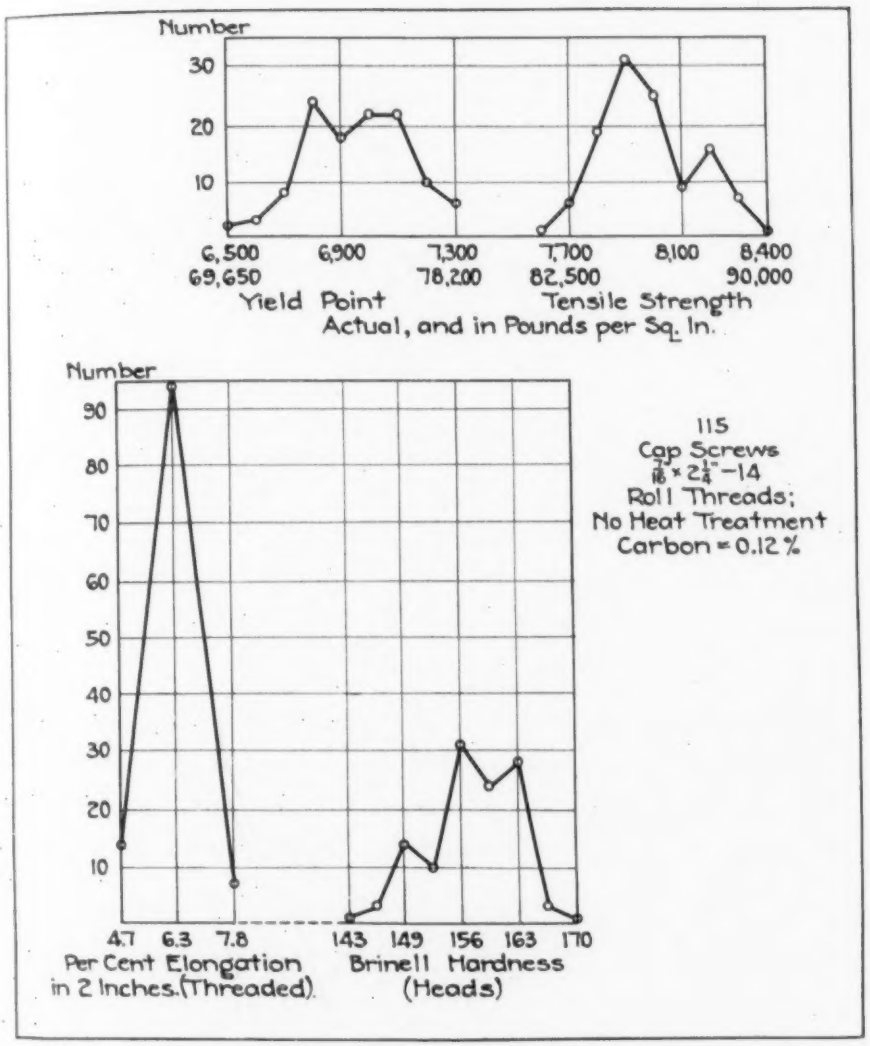


Fig. 2—Physical Properties of 115 Untreated Cap Screws, Carbon 0.12%.

deviations at both extremes. The average deviation from the most frequent is 1.6 units.

The Brinell results recorded in Fig. 1 were obtained by milling convenient lengths of the unstraightened wire to $\frac{9}{16}$ inch flats and making three rows of impressions, staggered, along the parallel surfaces.

The most frequent Brinell number is 137. The average deviation from the most frequent number is 5.0 units. It is obvious that the wire has flowed excessively to give the lowest figures; in other words, the impressions were made too near the edge.

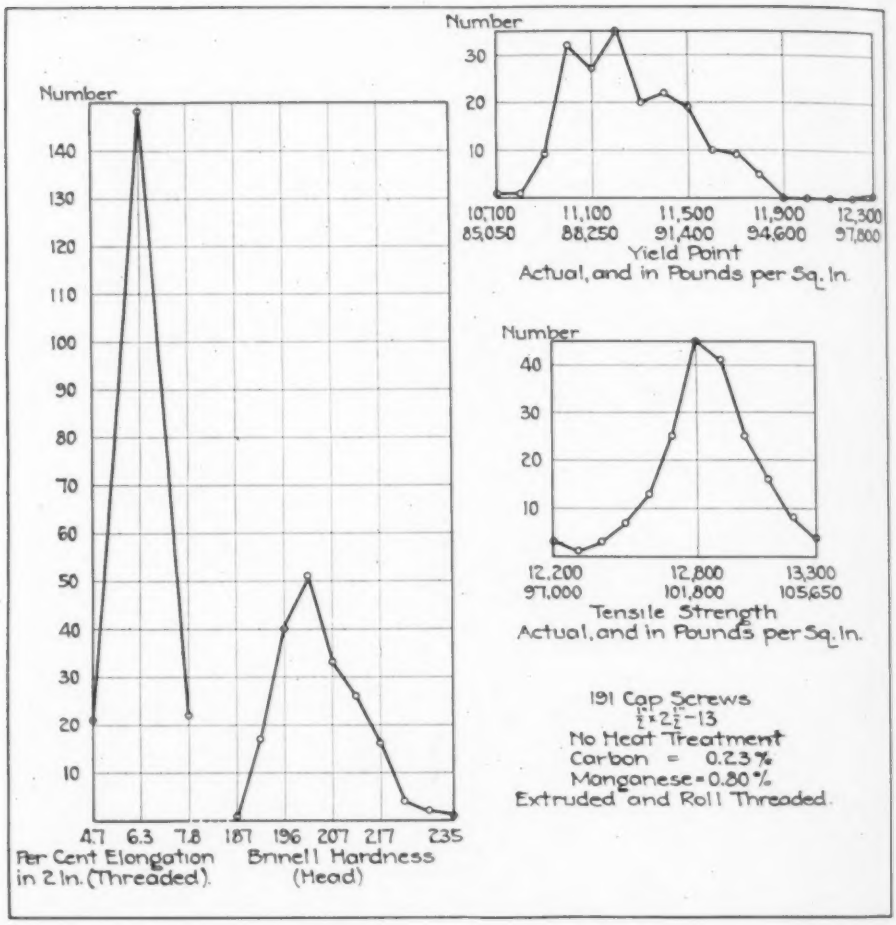


Fig. 3—Physical Properties of 191 Untreated Cap Screws, Carbon 0.23%.

But these results are typical of the distribution curves that will be found by numerous tests on any wire, rolled or drawn bar. A test on a 2-inch drawn hexagon bar gave a Brinell curve similar to the one in Fig. 1, except the few lower readings, so that the "tail" of the curve was shorter on that side. The bar was so wide that none of the impressions were too near the edge.

Thus we find that even in very simple cases the results of many similar tests fall into approximations of the normal or binomial distribution law. This calls for a most frequently occurring number, many numbers grouped about equally on both sides and diminishing to a few extremes to form the tails of the curve. Neither the mathematicians, the drawers of wire, or the manufacturers of bolts appear able to eliminate the tails of distribution curves.

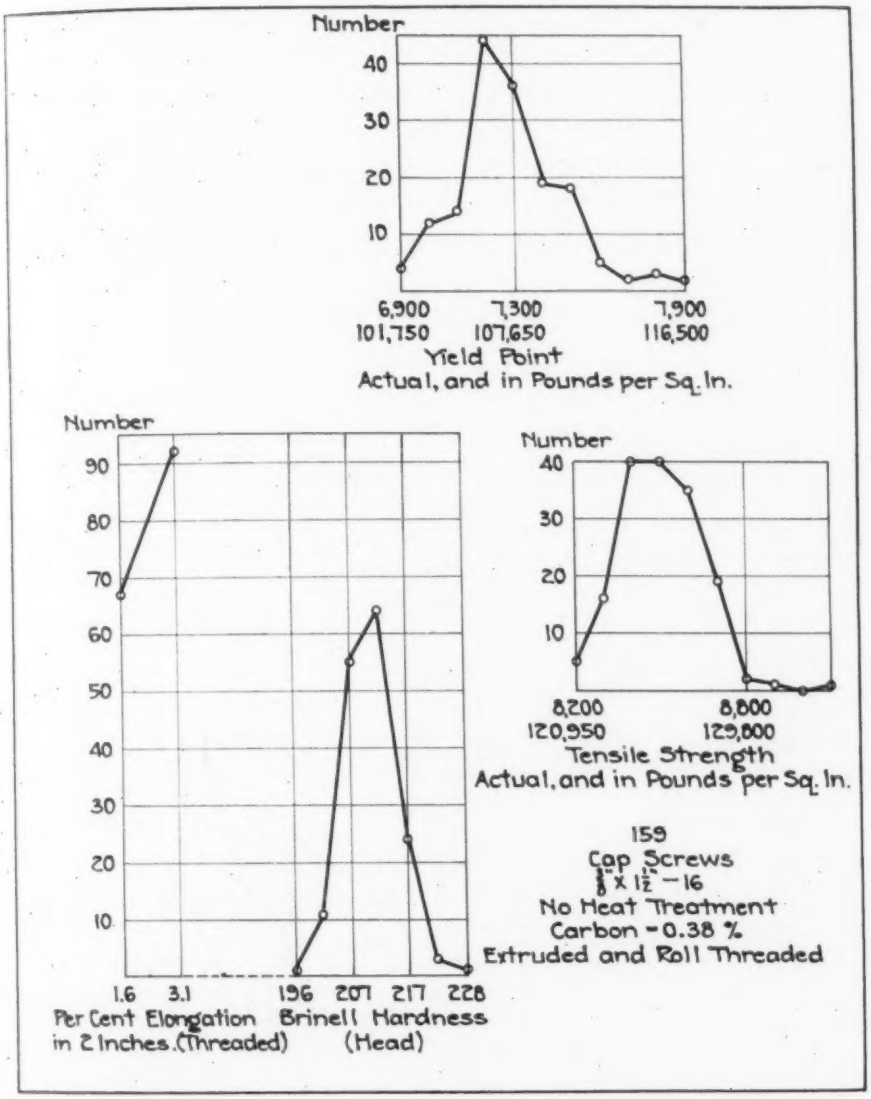


Fig. 4—Physical Properties of 159 Untreated Cap Screws, Carbon 0.38%.

Turning to the results of testing cap screws made in a very simple way from one coil of wire we may refer to Fig. 2. These cap screws were made by cold up-setting the head. At the same time the end to be threaded was extruded to pitch diameter so that later roll threading finished the essential processing of the screws. Trimming the heads to hexagon shape and pointing the threaded ends could in no way affect the physical properties.

The material used for the cap screws tested in Fig. 2 was the same as that used for the tests of Fig. 1, except its smaller size. A

longitudinal section of the wire at 1000 diameters is given in Fig. 15.

Here we have the properties of a low carbon steel as modified by the cold work of the roll threading process, and the strengthening effect of the threads. The wire had a tensile strength of some 65,000 pounds per square inch and a fracture strength of about 120,000 pounds per square inch. The cold working has not been carried far enough to cause brittleness, as indicated by the abundant elongation.

In the curves for yield point, tensile and hardness we see the irregularities consequent on the small number of tests, and in the Brinell curve the personal bias to read even divisions on the diameters may cause the saw tooth outline. The head hardness has no relation to the hardness of the threaded end; the upsetting has raised the head hardness some 20 or 30 points above the wire hardness.

The results indicated in Fig. 3 were made on bolts from wire containing 0.23 per cent carbon. Fig. 16 is a section of the wire at 1000 diameters. The higher carbon gives a pronounced effect.

The original wire is much harder than the 0.12 per cent wire and the effect of the cold working during extrusion and roll threading brings the mean tensile strength above 100,000 pounds per square inch.

A great many untreated cap screws are made of stock containing around 0.35 per cent carbon. Tests on extruded and roll threaded cap screws from one coil of wire containing 0.38 per cent carbon are recorded in Fig. 4.

The mean tensile strength is now around 125,000 pounds per square inch and the elongation is greatly diminished. The original wire had a tensile of around 85,000 pounds per square inch and a fracture strength of about 140,000 pounds per square inch. The product still has sufficient ductility for ordinary purposes. A typical longitudinal section through the wire is given in Fig. 17 at 1000 diameters.

In each of these charts on cap screws made without heat treatment the pieces were all cut off consecutively from the same coil of wire. Each lot was roll threaded simultaneously with the same setting of the threader. We shall not lengthen the discussion in explaining *why* the results appear as distribution curves instead of all with identical values. The facts are conclusive and easy to demonstrate by any investigator.

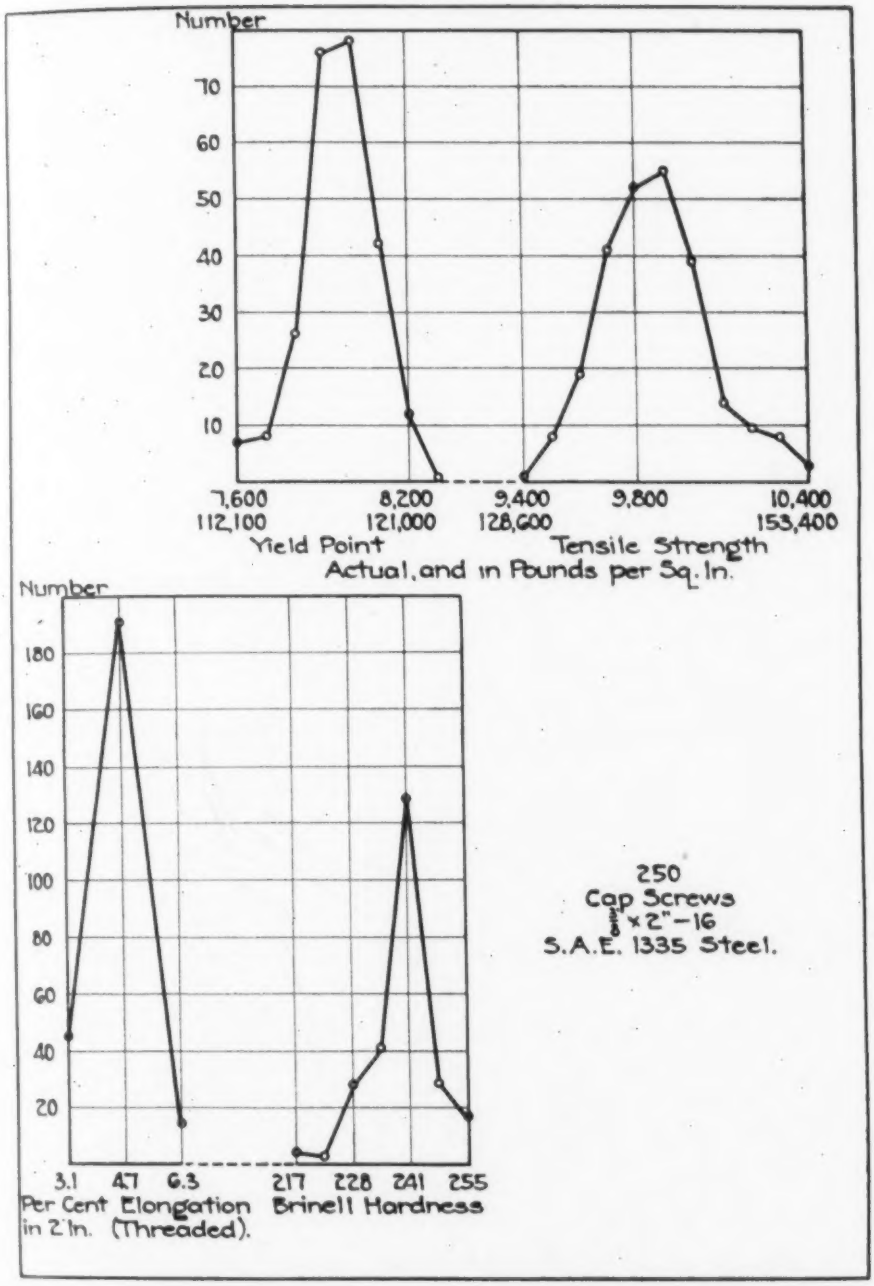


Fig 5—Physical Properties of 250 Treated Cap Screws, S.A.E. 1335 Steel.

If we now go to heat treated products and confine ourselves to cap screws made from a single coil of wire and treated in one lot we get distribution curves similar to the preceding.

Fig. 5 shows the results of testing 250 cap screws made of

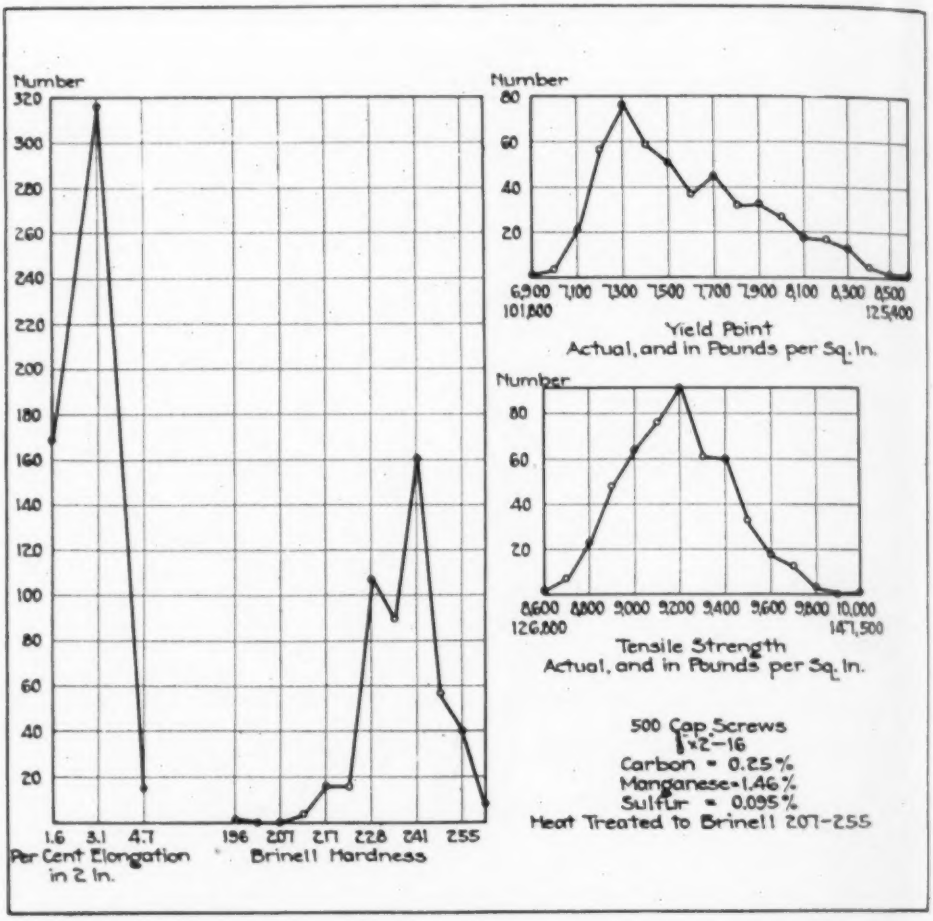


Fig. 6—Physical Properties of 500 Treated Cap Screws, Carbon 0.25%, Manganese 1.46%.

S.A.E. 1335 steel and treated to the range 207-255. Fig. 18 is a print at 1000 diameters showing typical structures after this treatment. The texture of the steel is much finer than the preceding and the properties are correspondingly better. The narrow range is rather significant.

Fig. 6 is of a steel with lower carbon and higher manganese, also treated to the same range, but showing much less elongation and considerably wider "spread." Of course a slightly wider "spread" would be expected from testing 500, rather than 250 pieces, but the difference is more than should result from this cause alone. Fig. 19 shows the typical microscopic structure at 1000 diameters.

Fig. 7 shows another series of 500 cap screws from the same lot as the preceding but drawn only to the range of 255-302. Fig.

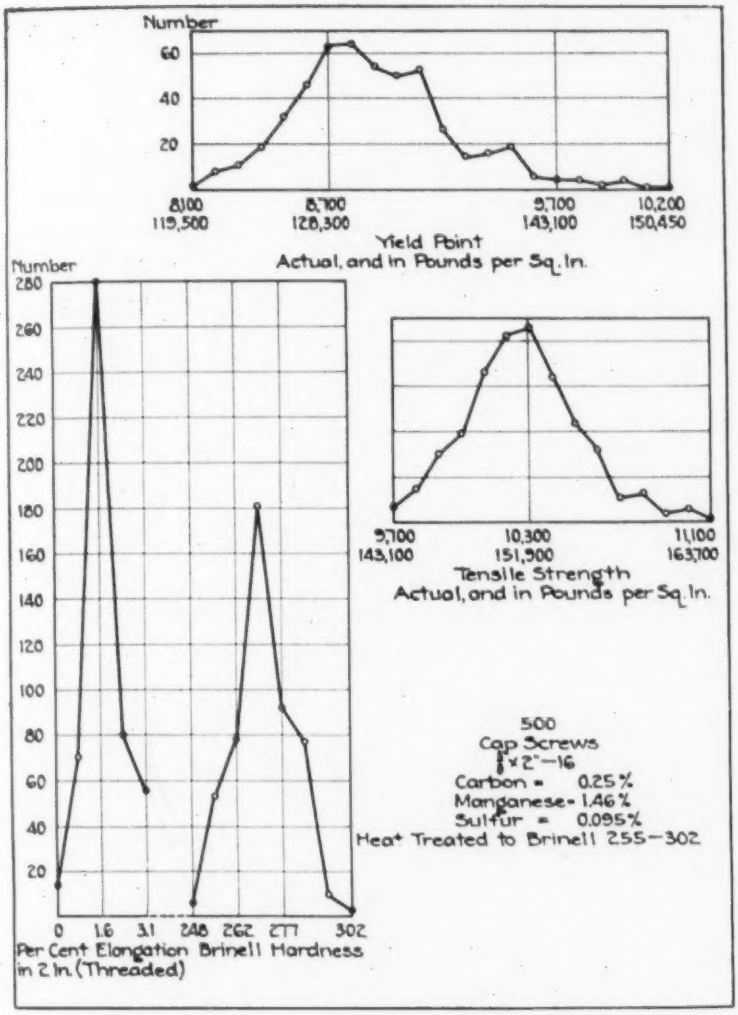


Fig. 7—Physical Properties of 500 Treated Cap Screws, Carbon 0.25%, Manganese 1.46%.

20 shows the microscopic structure, not appreciably different from the previous one.

Those who are not familiar with the precision of current heat treatment may be interested in the sharpness of the results. There is seen to be only slight overlapping of the results, even on testing 500 of each draw.

Fig. 8 records the results of testing 500 simple carbon cap screws of 0.36 per cent carbon content. Fig. 21 gives a typical microscopic section at 1000 diameters. The coalescence of the ferrite into rosettes has proceeded to considerable extent. This coalescence of the ferrite takes place easily and rapidly at the drawing temperature. With

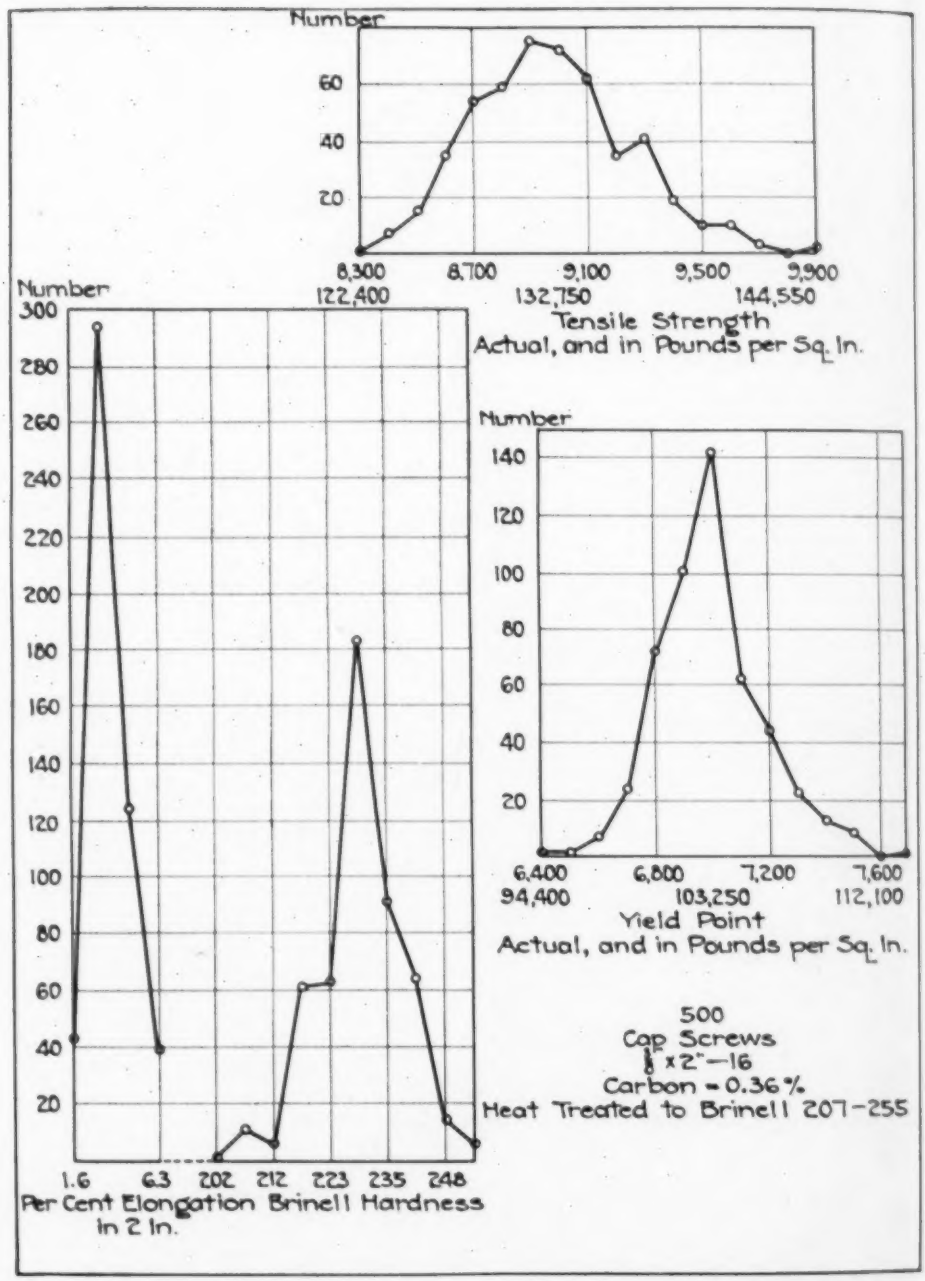


Fig. 8—Physical Properties of 500 Treated Cap Screws, Carbon 0.36%.

varying carbon content, as between different coils of wire held only within commercial ranges, a far greater "spread" in properties is to be expected.

But with the introduction of alloying elements such as nickel,

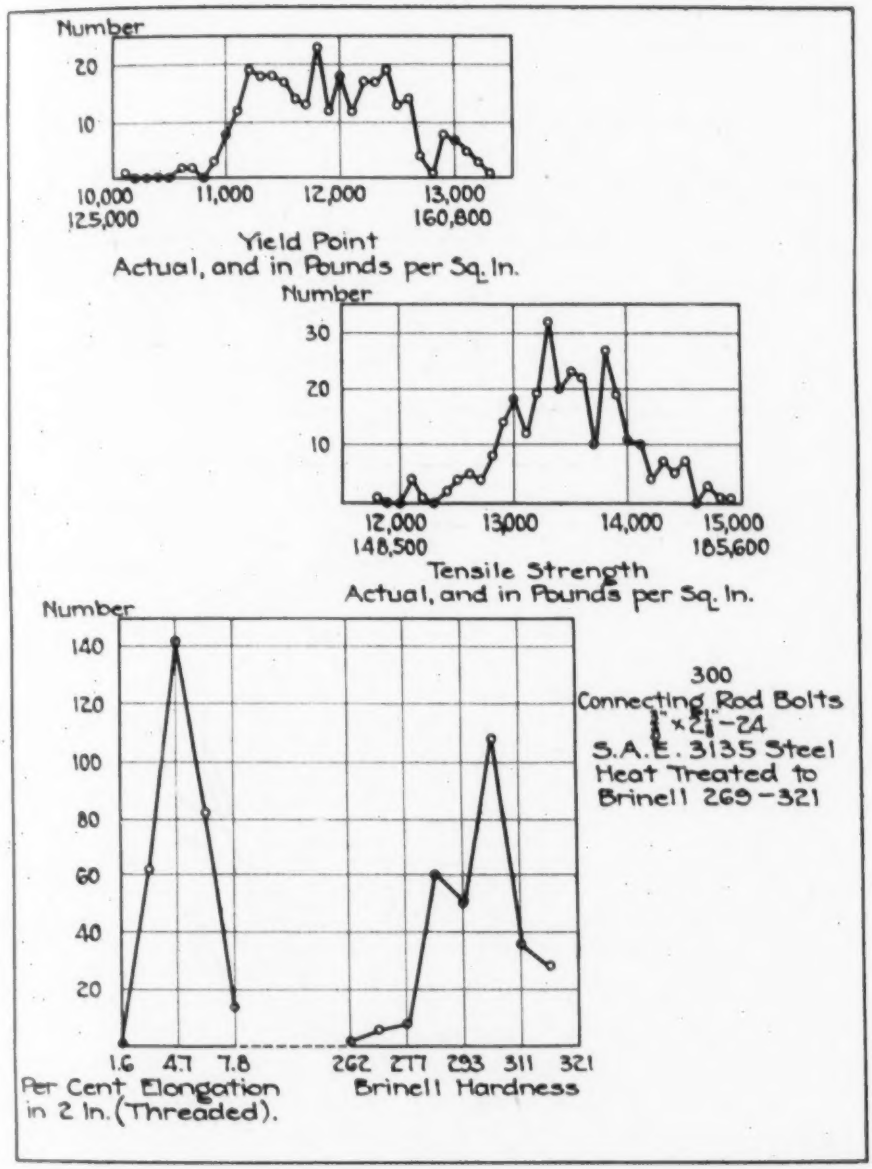


Fig. 9—Physical Properties of 300 Connecting Rod Bolts. S.A.E. 3135 Steel.

chromium and manganese, it is possible to hold to passably narrow ranges, even with commercial ranges of composition and many coils and many lots in the treatment.

In Fig. 9 are the results of testing 300 connecting rod bolts made from S.A.E. 3135 steel. They were picked at random from some 50,000 pieces as manufactured to Brinell range 269-321. The typical microscopic structure is shown in Fig. 22.

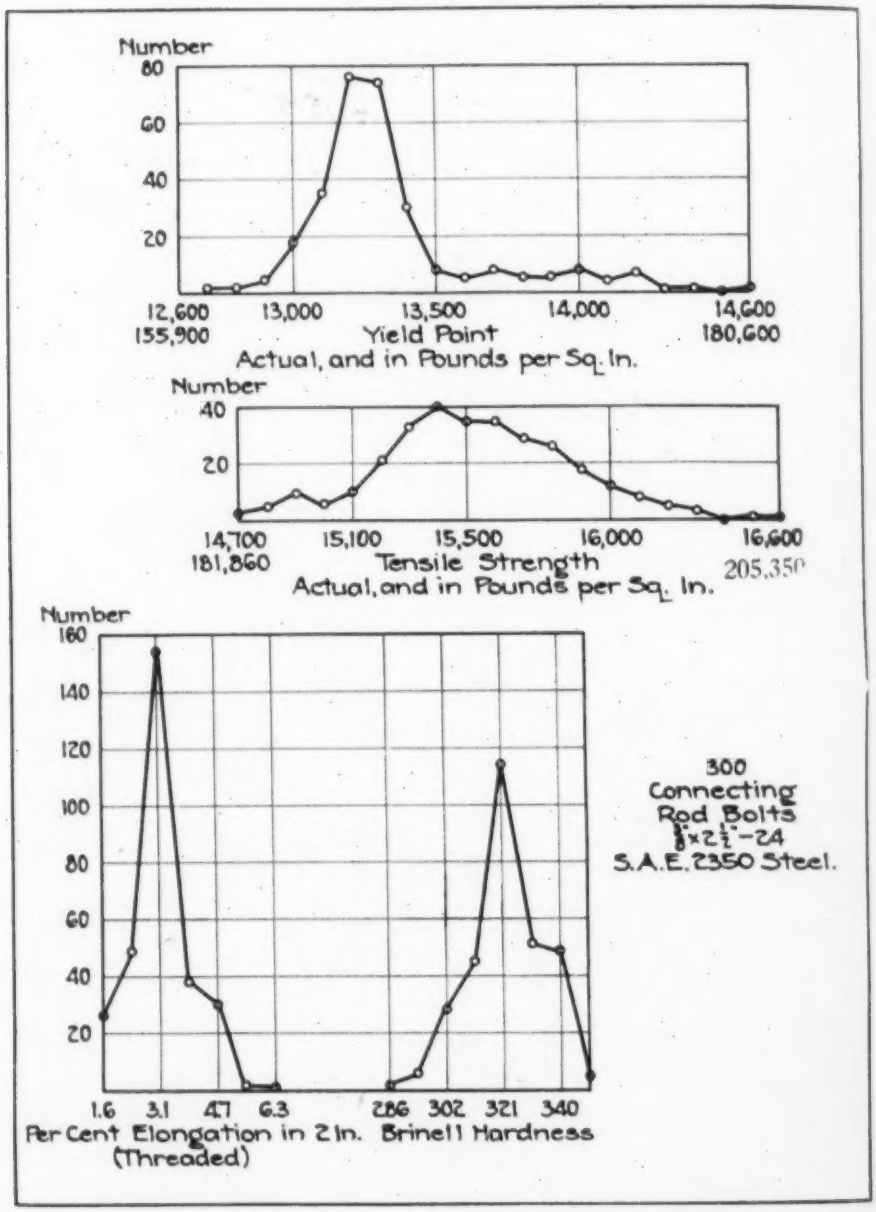


Fig. 10—Physical Properties of 300 Connecting Rod Bolts. S.A.E. 2350 Steel.

The cause of the strongly serrated form of the distribution might be placed on composition, dimensions, or treatment. Yet, that it is not necessarily so easily explained, is indicated by Fig. 10, the bolts for which were picked in a similar way from fully as many coils, batches and as large a number of manufactured pieces.

Fig. 10 is from tests on an S.A.E. 2350 steel and shows the

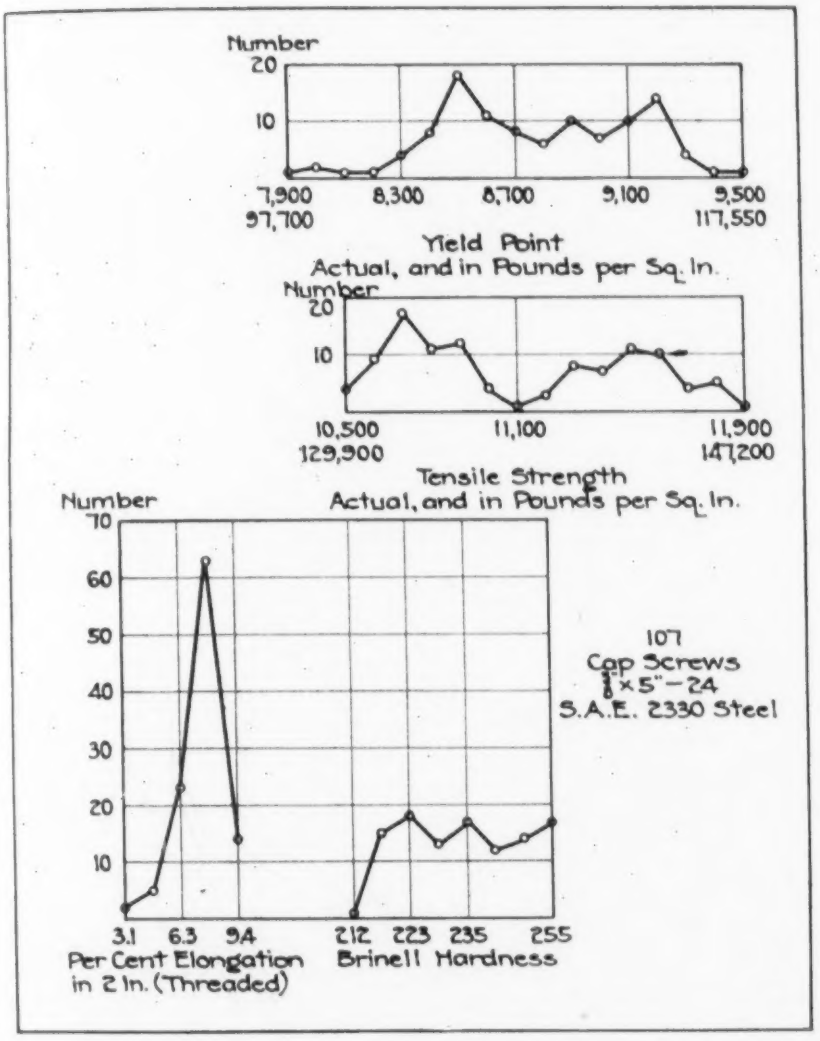


Fig 11—Physical Properties of 107 Treated Cap Screws. S.A.E. 2330 Steel.

results of attempting to withdraw the heats from the furnace with the yield point as near as possible to, but not below 13,100 pounds, actual. There is a heavy accumulation of results at 13,200 and 13,300 which leads one to suspect that knowledge of the requirement influenced the operator of the tensile machine. But, as a matter of fact, sample bolts actually were taken from the heat and pulled before the heat was withdrawn. Only when the samples showed a yield point as required was the lot taken out. If the first and later samples showed overdrawing or possibility of improvement, the lot was entirely rehardened and results discarded.

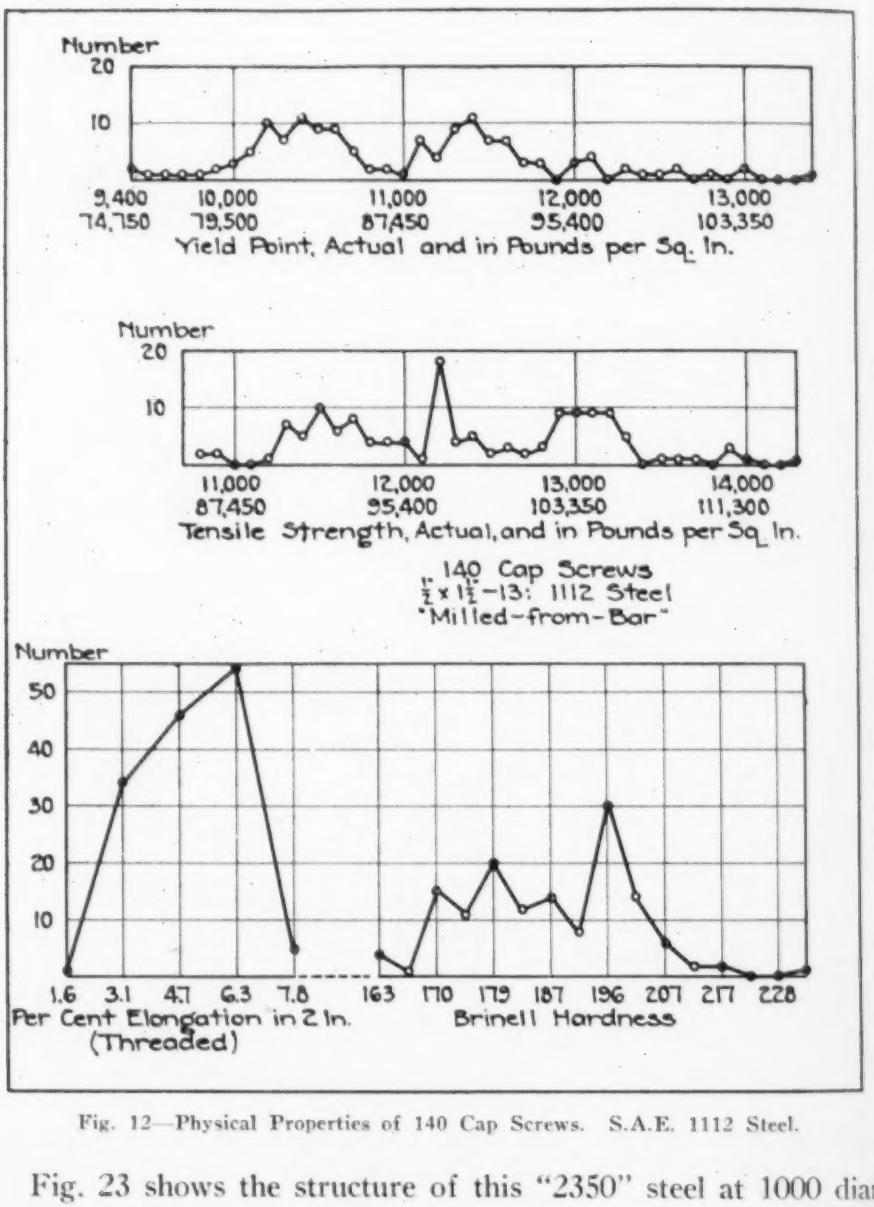


Fig. 12—Physical Properties of 140 Cap Screws. S.A.E. 1112 Steel.

Fig. 23 shows the structure of this "2350" steel at 1000 diameters as found in a bolt with mean properties.

Fig. 11 gives the results of testing 107 cap screws made of S.A.E. 2330 steel. The only requirement was that the bolts should have a minimum tensile strength of 125,000 pounds per square inch and a cold bend test of 180 degrees. The peculiarities of this lot are slightly accentuated by picking out of 10 pieces with Brinell hardness 241 for the thread test given later. The depression at Brinell 241 would accordingly be raised to a maximum of 22 at that point

but the ten results would be spread on the yield and tensile curves. No known reason can be offered for the shapes of the curves.

These 107 bolts were tested for hardness and laid out in groups for correlation of hardness with tensile strength. There were 17 bolts with Brinell 255 and none harder. Individual bolts of each hardness group had low coefficient of correlation as regards strength, but, by groups the average was as follows.

Brinell Number	Number of Bolts	Average Yield Point	Average Tensile Strength
212	1	99,000	129,900
217	15	105,050	132,400
223	18	104,400	133,250
228	13	106,400	134,250
235	17	108,600	136,600
241	12	110,350	139,450
248	14	113,700	142,750
255	17	113,100	144,000

In selecting a chart that will indicate the "spread" consequent on factors disturbing the uniformity of results there is a limit imposed by the width of the sheet. Astonishing ranges occur when low carbon bolts are quenched from the critical range, when untreated bolts are made from wire of varying hardness, when medium carbon bolts are carelessly treated or when even alloy bolts are non-uniformly treated in either quench or temper. Fig. 12 is a report on 140 "milled-from-bar" bolts that were expected to be uniform in properties yet, when actually tested, gave plots that are not too elongated. A typical longitudinal section of this S.A.E. 1112 steel is given in Fig. 25. Since these are untreated bolts the cause of the wide ranges is naturally laid to the varying hardness of the drawn bars from which the bolts were milled.

The data from the charts may be assembled in columns showing the mean tensile strength, in pounds per square inch, the full

Table I
Percentage "Spread" and Deviation of the Average

Material	Mean Tensile in Pounds Per Sq. In.	Full "Spread" of Tensile in % of Mean	Mean Tensile (Actual) Pounds	Average Deviation from Mean Pounds	Per Cent Deviation of Average
0.12% C	85,700	10.0	8,000	175	2.2
0.23% C	101,750	8.6	12,800	200	1.6
0.38% C	125,350	10.6	8,500	150	1.8
"1335"	146,000	10.1	9,900	200	2.0
1.46% Mn	135,700	15.2	9,200	225	2.4
1.46% Mn	151,900	13.6	10,300	240	2.3
0.36% C	132,750	17.8	9,000	252	2.8
"3135"	167,000	23.0	13,500	450	3.3
"2350"	191,750	12.3	15,500	295	1.9
"2330"	137,300	12.6	11,100	375	3.4
"1112"	98,100	28.3	12,350	700	5.7

"spread" of the range in per cent of the mean tensile strength, the actual mean tensile strength, the average deviation from this mean, and the average deviation in per cent of the mean. In this way we get a measure of the full range and also a measure of the flatness of the distribution curve. Obviously, the larger the percentage deviation from the mean the flatter the curve. (See Table I).

The Strengthening Effect of Threads

Since the strength of bolts in pounds per square inch is commonly calculated on the root diameter of the threaded section the quantity is in error by an amount equal to the strengthening effect of the threads. This assumes a straight cylindrical section 2 inches long as a standard section.

The subject is a rather complicated one. There is the necessity of fracturing across one or more thread crests. There is the well-known strengthening effect of greatly shortened test section. In untreated bolts we have the cold working of roll threading, fine or coarse threads. Then, there is the cold working of the threaded section to pitch diameter before roll threading.

In untreated bolts we may arrange the strengthening effect in the following sequence:

1. Cut threads, fine
2. Cut threads, coarse
3. Milled and fine roll threads
4. Milled and coarse roll threads
5. Extruded and fine roll threads
6. Extruded and coarse roll threads.

This sequence was carried out on $\frac{7}{16}$-in. blanks from a coil of wire with 0.36 per cent carbon with average results as follows:

	Yield Point Lb. Per Sq. In.	Tensile Strength Lb. Per Sq. In.	Elong. in 2" %
Original wire (3)	74,500 (indistinct)	87,000	17.0
Fine cut threads (20)	66,050	91,750	6.5
Coarse cut threads (20)	75,000	92,100	4.7
Milled, fine roll threads (20)	78,900	93,600	6.3
Milled, coarse roll threads (20)	77,100	98,600	5.5
Extruded and fine roll threads (20)	82,600	102,800	4.7
Extruded and coarse roll threads (20)	99,650	112,500	4.1

The irregularities of the results indicate the complexity of the effect of threads. Doubtless the structure of the material and its reaction to working and pulling influences the results.

The cold working and plastic flow of the metal at the root of

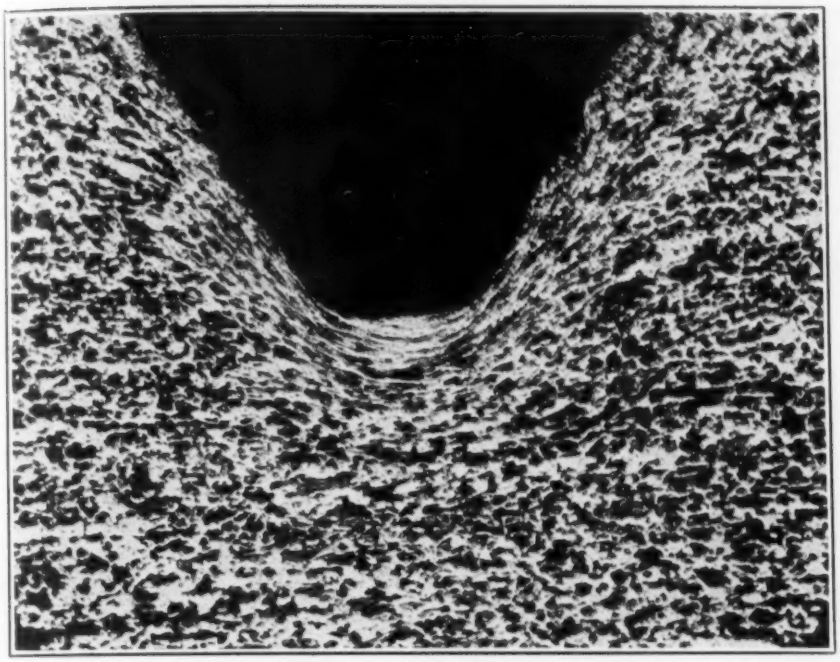


Fig. 13—Section Through Root of Coarse Roll Thread, ×100. Untreated $\frac{7}{16}$"x2" Cap Screw, Extruded and Roll Threaded. Carbon = 0.36.

a coarse roll thread is shown in Fig. 26 at 100 diameters. The metal is heavily deformed, but not torn or ruptured, at the very root of the thread. The effect penetrates much further than the heavy deformation. The result is a general and desirable stiffening and strengthening of the weakest section of the bolt.

Since wire with 0.12 per cent carbon strengthens on pulling to a fracture load of some 125,000 pounds per square inch and wire with 0.35 per cent carbon strengthens to a fracture load of about 145,000 pounds per square inch, we may expect these amounts to establish approximate limits to the cold-working effect in the threading of bolts. There is added to this the stiffening and strengthening effect of thread ridges and short test section.

It appears possible to obtain practically all of the stiffness and strength of heat treated bolts by cold working alone. But at the same yield point and tensile strength, the heat-treated bolts have much greater elongation. In other words, they are far less brittle.

Another limitation to effective thread strengthening is the small difference between root area and full stem area. In other words, the threaded section may be strengthened so that the bolt fails in the main stem section. This sometimes happens in commercial bolts.

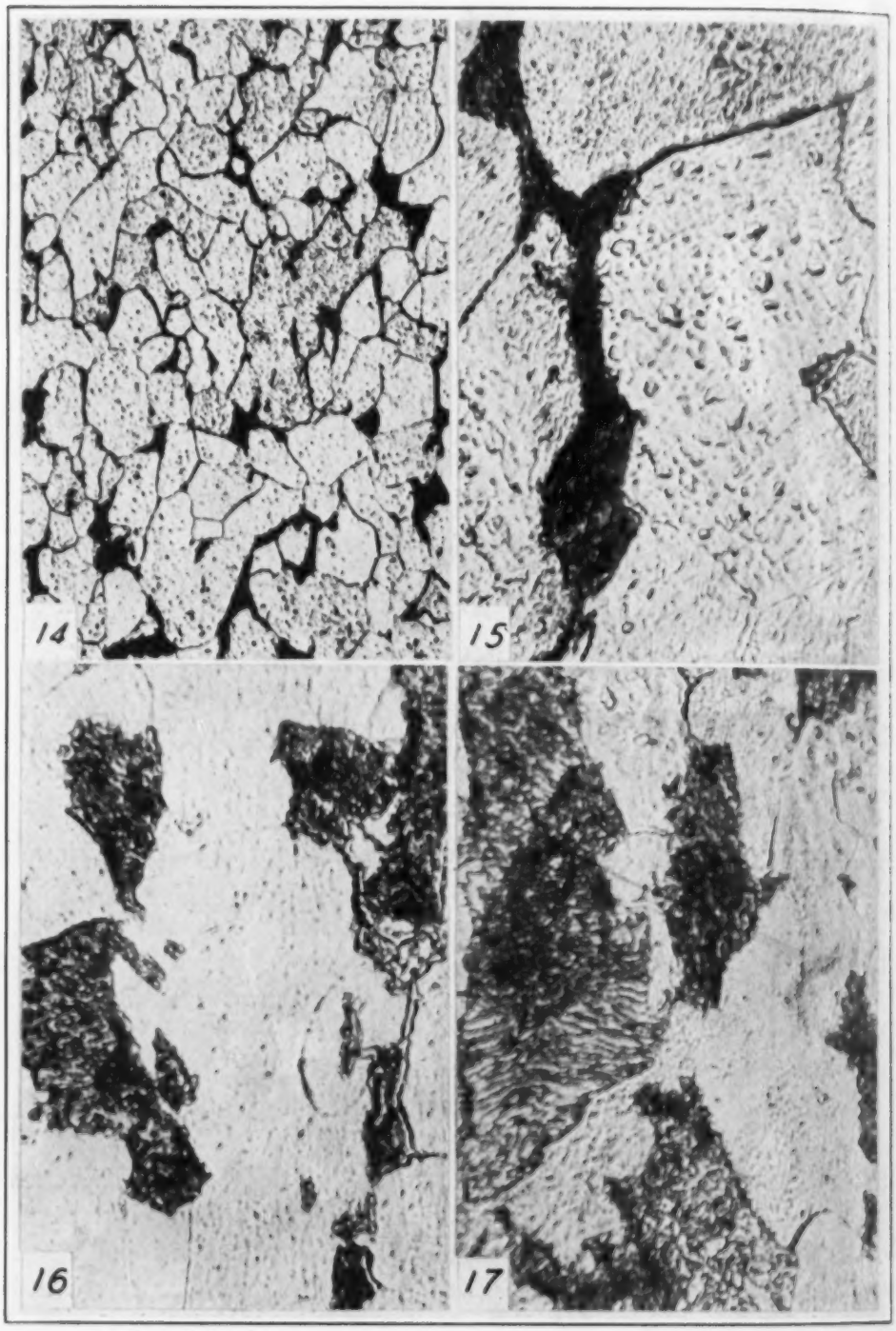


Fig. 14—Longitudinal Section Through 0.730 inch Steel Wire, ×200. Carbon = 0.12 Per Cent. Brinell 137.

Fig. 15—Longitudinal Section of 0.425 inch Steel Wire, ×1000. Carbon = 0.12 Per Cent. Brinell 137.

Fig. 16—Longitudinal Section of 0.490 inch Steel Wire, ×1000. Carbon = 0.23 Per Cent. Brinell 143.

Fig. 17—Longitudinal Section of 0.365 inch Steel Wire, ×1000. Carbon = 0.38 Per Cent. Brinell 160.

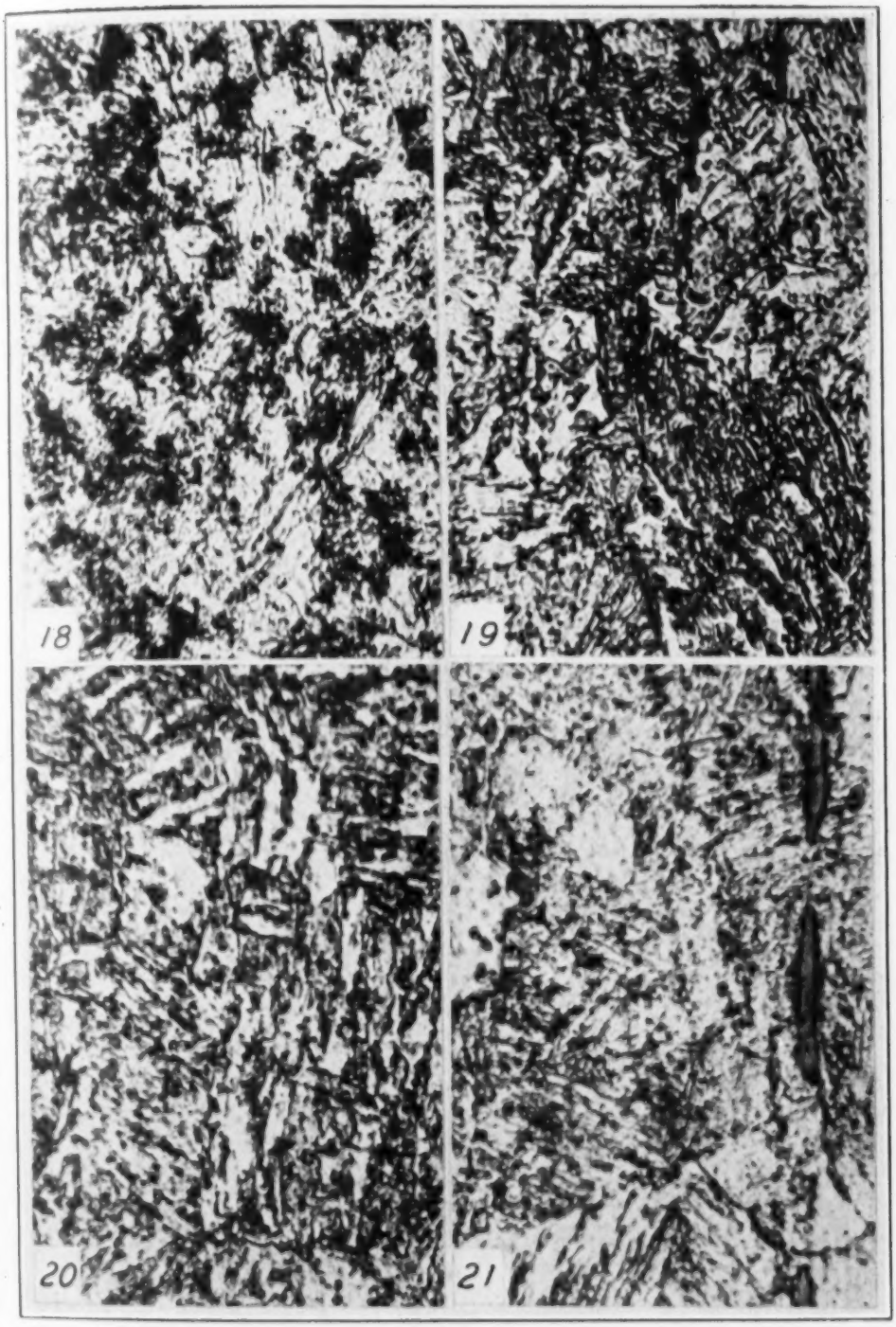


Fig. 18—Longitudinal Section of Heat Treated Cap Screw Made of S. A. E. 1335 Steel. × 1000. Brinell 241.

Fig. 19—Longitudinal Section of Heat Treated Cap Screw, ×1000. Carbon = 0.25 Per Cent; Manganese = 1.46 Per Cent. Brinell 241.

Fig. 20—Longitudinal Section of Heat Treated Cap Screw, ×1000. Carbon = 0.25 Per Cent; Manganese = 1.46 Per Cent. Brinell = 286.

Fig. 21—Longitudinal Section of Heat Treated Cap Screw, ×1000. Carbon = 0.36 Per Cent. Brinell 228

Between the sizes of ¼ and ¾ inches, fine threads, the full stem area is only from 51 to 26 per cent greater than the root areas. Any size having the threaded part strengthened more than its corresponding area increase can utilize only the fractional area increase. Further strengthening is wasted.

But there is another phase of the strengthening effect of threads that relates to heat treated bolts alone. With the entire bolt of the supposedly uniform micro-texture after treatment, just how much stronger is the bolt than a test piece whose diameter is the same as the root diameter? This assumes that the strength per square inch has been figured from the root diameter.

In order to obtain some empirical information of this question eight lots of twenty bolts each were divided, half being tested as normal bolts, the other half being made into test pieces of root diameter before the testing. In some cases the machining was done before heat treatment, in the other cases after.

Twenty cap screws, ½ inch x 3¾ inch—20 (fine threads), of 0.10 per cent carbon content, had ten pieces machined to root diameter for a 2 inch length. All of the bolts were then quenched from 1700 degrees Fahr.

Another lot of cap screws made from the same stock (0.10 per cent carbon), ¾ inch x 4¾ inches—10 (coarse threads), had ten pieces turned down to root diameter for 2 inches. All pieces were then quenched from 1700 degrees Fahr. in water. Fig. 26 shows the typical structure of this low carbon steel as quenched from 1700 degrees Fahr.

Twenty cap screws, ½ inch x 4½ inches—13 (coarse threads), made from steel containing 0.41 per cent carbon, were quenched and tempered. The Brinell hardness of each was determined and the lot divided as equally as possible. One set of ten bolts was then turned down to root diameter for 2 inches.

Another lot of cap screws made from straight carbon steel, 0.38 per cent, ⅝ inch x 4$\frac{11}{16}$ inches—18 (fine threads), had ten pieces machined to root diameter before the entire lot was heat treated.

Ten cap screws of Brinell hardness 241 were taken from the lot tested according to Fig. 12 before those cap screws were tested. The ten pieces were machined to root diameter—necessarily after the heat treatment. This lot was of 2330 steel, ⅜ inch x 5 inches —24 (fine threads).

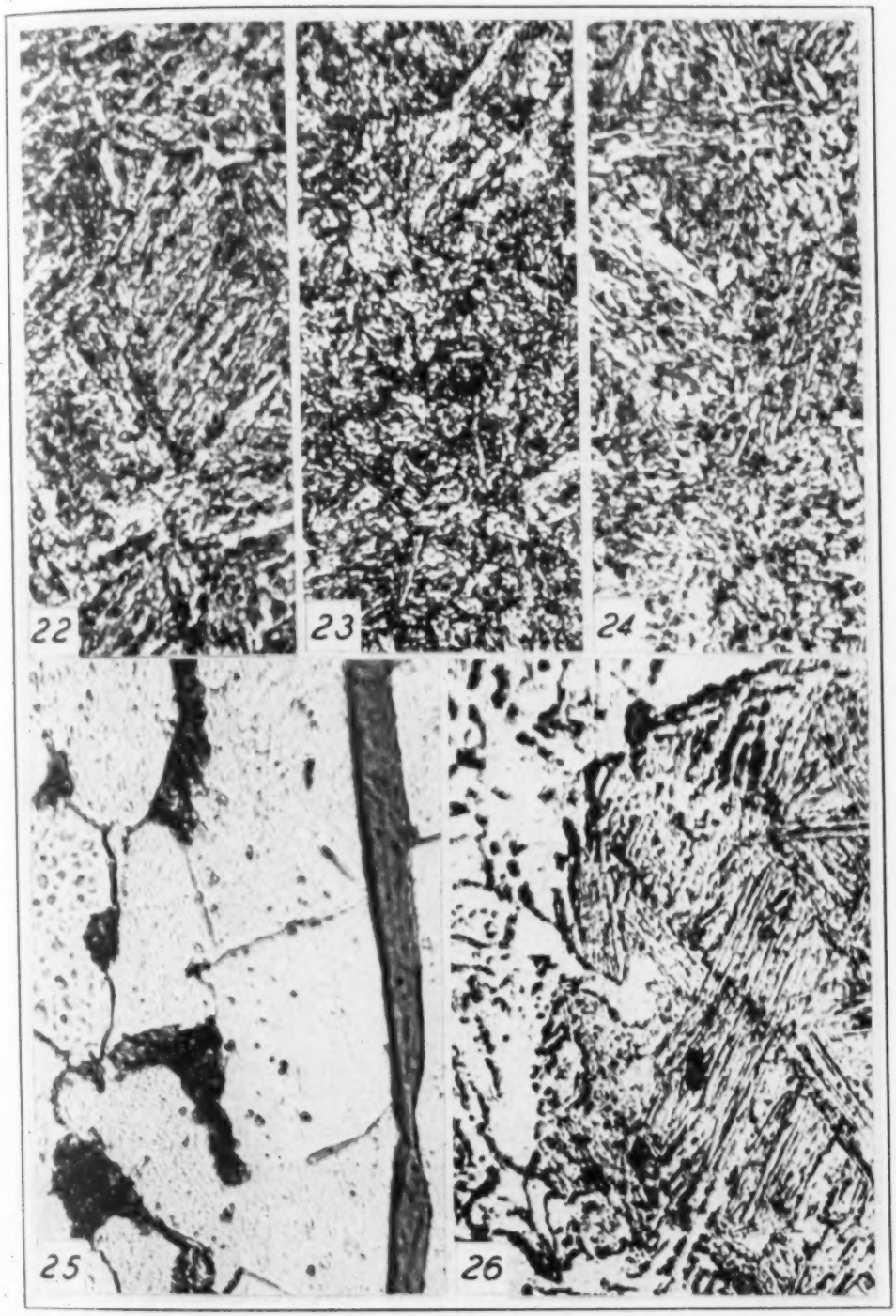


Fig. 22—Longitudinal Section of Heat Treated Connecting Rod Bolt, ×1000. S.A.E. 3135 Steel. Brinell 302.

Fig. 23—Longitudinal Section of Connecting Rod Bolt, ×1000. S. A. E. 2350 Steel. Brinell 321.

Fig. 24—Longitudinal Section of Heat Treated Cap Screw, ×1000. S.A.E. 2330 Steel. Brinell 241.

Fig. 25—Longitudinal Section from Cap Screw Made by "Milled-from-Bar" Pieces. S. A. E. 1112 Steel, ×1000. Brinell 179.

Fig. 26—Longitudinal Section of Quenched Low-Carbon (0.10 Per Cent) Cap Screw, ×1000 Brinell 163.

Table II
Normal Bolts vs. Test Bolts

Material and Size		Av. Brinell	Av. Yield	Av. Tensile	Av. Elong. %	Inc. in Yield by Threads %	Inc. in Tensile by Threads %	Inc. in Elong. by Test Section %
0.10% C ½"	f	198	73,000	107,900	9.4	23.0	23.9	.....
		192 b	59,400	87,100	15.5	....	....	64.9
0.10% C ¾"	c	157	70,340	94,400	14.1	11.8	10.6	.....
		164 b	63,020	85,330	23.6	....	....	67.4
0.41% C ½"	c	238	101,280	125,290	5.3	26.6	16.3	.....
		235 a	79,970	107,720	17.9	....	....	238.
0.38% C ⅝"	f	252	112,450	133,660	11.9	18.5	14.2	.....
		252 b	94,910	117,080	18.9	....	....	58.8
"2330" ⅜"	f	241	110,480	139,550	8.3	25.8	21.3	.....
		241 a	87,840	115,060	19.7	....	....	137.
"3135" ½"	f	295	140,090	155,970	9.4	6.2	5.9	.....
		295 a	131,340	146,820	17.2	....	....	83.0
"2350" $\frac{9}{16}$"	f	340	156,200	172,400	2.2	18.2	13.0	.....
		340 a	127,700	151,000	14.5	....	....	552.
"2350" ⅝"	f	342	166,820	181,280	2.4	19.7	12.8	.....
		339 b	139,410	160,700	15.5	....	....	546.

Another lot of cap screws made from 3135 steel, ½ inch x 3¾ inches—20 (fine threads), was heat treated and then divided as equally as possible according to Brinell reading on the head before ten pieces were turned down to root diameter.

Finally, two lots of cap screws made from 2350 steel were used. One lot had pieces $\frac{9}{16}$ inch x $3\frac{3}{32}$ inches—18 (fine threads) and ten of them were turned down to root diameter *after* heat treatment.

The other lot of 2350 steel had dimensions ⅝ inch x $3\frac{3}{16}$ inches —18 (fine threads) and ten pieces were machined to root diameter *before* heat treatment.

In Table II the results are summarized. The "f" or "c" after the bolt diameter indicates the fine or coarse threads. The normal bolts are given first and the test bolts second. The "a" or "b" at the Brinell of the test pieces indicates the machining after or before heat treatment. In all cases the quantities are the averages of the ten pieces in each half lot.

With exception of the low carbon steel, all of the microscopic structures are similar to those of the charts and are not repeated.

In general, the larger and stronger the bolt the less the strengthening effect of the threads. Evidently the data are far too scanty to justify precise conclusions. But the possibility of correlating the strengthening effect with the pitch area, or any intermediate area, appears rather distant. This conclusion will be more clear from Table III, which gives the increase of pitch area over root area for each lot.

Table III
Deviations of Bolts vs. Test Pieces

Material and Size	Excess of Pitch Area over Root Area	Inc. in Tensile by Threads	Average Deviation from Mean Normal Bolts		Test Bolts	
			Yield	Tensile	Yield	Tensile
	%	%	%	%	%	%
0.10% C ½″ f	15.5	23.9	5.1	4.9	2.7	3.4
0.10% C ¾″ c	21.9	10.6	5.7	5.2	3.2	4.1
0.41% C ½″ c	26.3	16.3	3.4	3.3	3.2	1.3
0.38% C ⅝″ f	13.5	14.2	7.6	4.2	4.2	2.3
"2330" ⅜″ f	17.4	21.3	2.4	2.4	1.7	1.7
"3135" ½″ f	15.5	5.9	1.5	1.1	0.9	0.3
"2350" ⅜″ f	15.2	13.0	2.3	1.8	2.3	1.5
"2350" ⅝″ f	13.5	12.8	1.5	1.4	0.6	0.6

Although each of these small lots of only ten pieces contains far too few members to justify final proof, one is immediately impressed by the smaller average deviation of the machined down bolts. The small average deviation of the alloy bolts confirms the previous statement about better uniformity in the bolts made from alloy steel.

Summary

It is clearly perceived that this brief record of the properties of some of the commercial bolts now being made contains no intimation of finality. There is rapid progress in the industry. Alloy bolts are more and more extensively used. The requirements in physical properties and uniformity and reliability are always more exacting.

It is hoped that the simple distribution charts will give a clearer idea of the possibilities and limitations peculiar to threaded sections. The improvements in furnaces and knowledge of physical properties leads one to believe that finer properties and better uniformity can soon be attained.

In reviewing the evidence, a very important conclusion pertains to the effect of the threads compared with a smooth test section. In a tensile test the external spiral of steel outside of the root area has a moderate stiffening and strengthening effect but its effect in decreasing elongation and reduction of area may be much greater. While this spiral more or less succeeds in holding the metal from stretching and contracting the tension builds up until finally the metal parts with a violent rupture. There is far more localization of fracture than in the straight 2-inch length of a standard test section. Small details of structure and dimension may

be expected to play a larger part than in a 2-inch length where equalization, longer plastic flow and a much reduced rupture load are all in play. In the tensile test of a bolt the maximum load and fracture load may be very close together. This infers that the maximum load on a bolt partakes distinctly of that uncertainty and wide fluctuation characteristic of fracture tensions.

This means a great deal to those interested in the properties of bolts. It forces us to the conception that any given material in the form of a threaded bolt will show a greater "spread" in elastic, yield, and tensile properties than the same material in the form of standard test sections. Greater allowance must be made in specifying limits. There is an obvious futility in placing minima and maxima, in specifying restricted ranges in hardness, yield points, tensile strength and impact, or other requirement. This futility is only the more delusive if the specifications are taken from charts or tables derived from standard test pieces and indiscriminately applied to bolts.

For production work the most practicable control test is by the Brinell hardness machine. With this control the 50-point range serves well as a working basis. But the results of sampling and testing should be interpretated with a knowledge of the probable distribution of results.

But bolts are intended primarily to be used as tension members in some sort of construction. A minimum yield point or tensile strength gives a practical assurance of the required suitability. The tensile testing machine thus completes the report on a bolt.

But, if a minimum is specified, the manufacturer must be allowed his range in tensile properties. It is futile to specify a minimum and expect the average or mean of many bolts to be found only a very few hundred pounds above. It may be possible to have an average deviation only three or four hundred pounds from the mean value, but the extreme values will probably cover a range of several thousand pounds. The type of equipment used in treating bolts and the care in operation can maintain a certain uniformity. But no evidence at hand indicates that "specifications" have anything to do with the characteristics of distribution curves.

As to the improvements and possibilities in developing better bolts, comparatively rapid progress is being made. It is well known that the mechanical equipment for the manufacture of bolts is rapidly changing to provide greater speed and accuracy.

Heating furnaces and quenching tanks are improving even more rapidly. The automatic control of temperature, the circulation of furnace atmospheres and uniform heating, and the stirring of the quenching bath are all being perfected.

The sampling, analysis and testing of wire, bars and finished product is becoming more frequent and thorough.

The old problems that originate in the steel ingot itself are very difficult for the bolt maker to solve. The structure of steel ingots is a very important source for that "spread" that appears in our distribution curves. There would be distribution curves even if ingots were perfect, but it is obvious that segregation, banding, inclusions, secondary pipes and seams are prolific sources of inequality in the final properties of bolts.

It is now known that annealed wire of as high as 0.55 per cent carbon is not difficult to cold head and roll thread. It may be either simple carbon or alloy stock. The use of the higher carbon ranges allows tensile strengths up to 175,000 pounds per square inch in simple carbon steels and up to or above 200,000 pounds per square inch in alloy steels. Even at these strengths and with hardness from 300 to 400 Brinell there may be from 1.5 to 6.0 per cent elongation in the threaded bolt. The 2350 bolts of Fig. 10 and the thread test series, at 350 Brinell, will bend cold through 180 degrees without fissuring.

Accordingly, if finer bolts are needed, they are pretty sure to be supplied. And they will be provided with a uniformity in properties that was entirely unknown a few years ago.

DISCUSSION

Written Discussion: By H. W. Maack, assistant chief chemist and metallurgist, Crane Co., Chicago.

The author's probability curves plotted from tests of hundreds of bolts make an interesting study, particularly for bolt users and manufacturers. His distribution curves from results of tests of more than a hundred bolts from a single coil of wire, lead one to conjecture on the probable reasons for the spread in physical properties, whether due entirely to nonuniformity of structure of the wire or partly to variations in bolt manufacture.

The author reports elongation in 2 inches although many of the screws tested are shown to be only 2 inches long. Was elongation measured over a gage length of four times the root diameter as is customary for sections not of standard size?

Referring to the strengthening effect of threads, we found several years

ago in connection with some tests of various forms of bolt studs for severe service conditions, that a die-cut or single lathe tool-cut thread added about 10 per cent to the strength over that of a plain specimen turned down to root diameter. The stud used was heat treated alloy steel, ¾ inch in diameter. Heat treatment was done before machining. The increase in strength mentioned is within the range of results reported by Mr. Pulsifer but much lower than the average of his tests. The difference, no doubt, is due to the methods of forming the threads, rolling undoubtedly increasing the strength. Our tests showed no increase in strength by die or lathe cutting as shown by testing after machining-off such threads. Ratio of area of thread cross-section to root of thread cross-section also is a factor.

Threads on a bolt have about the same effect on elongation as a series of notches, localizing the stretch to the thread roots. It is important that this effect be understood in evaluating the results of tests of threaded sections, as is pointed out by Mr. Pulsifer.

Written Discussion: By C. L. Harvey, metallurgist, Lamson and Sessions Co., Kent, Ohio.

Before discussing the data on "The Physical Properties of Fine Bolts" submitted by Mr. Pulsifer, I should like to congratulate him upon its preparation.

In spite of the fact that many, many millions of bolts are used annually, very little work has been published regarding the range of physical properties that may be expected from any given type of material.

In presenting such data, I believe that the statistical method is highly desirable, but I would suggest that percentages of the whole be used rather than the actual number of specimens tested. I should also like to emphasize the desirability of "no discards." Producers and consumers alike are not troubled by the most frequent values obtained on a given specification, but they are troubled by the small percentage of material running close to, or just outside the limits of the specification; in other words, the extremes on the probability curve.

I further agree with Mr. Pulsifer that, regardless of the care used in selecting material and making it into bolts, a probability curve will be obtained showing most frequent values, a smaller number of adjacent values, and a very few extreme values. The general shape of this curve cannot be changed, but its "spread" can be altered by closer chemical limits, by more uniform heat treatment or by the addition of alloying elements.

The narrowing down of chemical limits must be treated cautiously. In special cases it can be done, but it usually means delay in obtaining material and sometimes extra cost. It is doubtful at the present time if the mills would be prepared or willing to guarantee narrower ranges for the entire bolt industry.

The use of alloying elements to develop higher physical properties and greater uniformity is coming into quite general use in many industries but it entails additional cost, and for that reason is not always desirable.

The question of more uniform heat treatment has always been a serious problem for the bolt maker and it has only recently been solved by the furnace designers. It is no easy task to design equipment capable of uniformily heating and quenching millions of small parts daily. However, suitable equipment

is now available from several manufacturers which leaves little to be desired as far as uniformity of product is concerned.

I quite agree with Mr. Pulsifer as regards the spread of values on bright bolts from any one type of material, but I feel that he has not given the heat treated straight carbon material its just dues. I cannot help but feel that the wide spread of results is due to the type of equipment rather than to the material used. In proof of this I offer the following:—

A bundle of 0.18 per cent carbon—0.79 per cent manganese wire was picked at random from stock and made into ⅜ x 1¼-16 cap screws by the cold upset and roll-thread process. One group of these bolts was tested without heat treatment; a second group was very carefully treated in the laboratory; a third group was put through the regular production line along with other bolts. The production unit was of the continuous travelling hearth type, automatically controlled. The treatment was one commonly used in the industry for removing heading strains in the lower carbon materials; viz., tempering at about 1000 degrees Fahr. The results of this test are shown below:—

		Brinell	Yield Pt.	Ult. Str.
Bright	Bolts—Range	179-196	.....	95,900-99,600
	Spread	17	.....	3,700
Lab.	Treated—Range	131-156	72,600-78,800	82,500-87,900
	Spread	25	6,200	5,400
Prod.	Treated—Range	143-163	75,600-82,600	86,800-92,400
	Spread	20	7,000	5,600

This shows that at tempering temperatures the production unit gives as uniform results as can be obtained in the laboratory under very carefully controlled conditions.

A bundle of 0.30 per cent carbon—0.66 per cent manganese material was then taken from stock and made into ⅜ inch x 1½ inch-16 cap screws. One group of these bolts was tested bright; a second group was heat treated and tempered in the production unit already referred to, along with other cap screws. The results are shown below:

		Brinell	Yield Pt.	Ult. Str.
Bright	Bolts—Range	196-212	.....	105,400-115,700
	Spread	16	.....	10,300
Heat	Treated—Range	196-241	105,900-113,200	118,800-127,600
	Spread	45	7,300	8,800

Comparing these results with the results shown on pages 7 and 12 of Mr. Pulsifer's paper, we find that the bright bolts have approximately the same range, but the bolts heat treated in the new type equipment show a spread in yield point and ultimate strength of only about 40 per cent of the author's.

The spread in Brinell hardness on the head appears to have little significance in any of the results.

These data, I think, show the improvement in uniformity obtainable with equipment now on the market.

In conclusion, I again want to emphasize the three methods by which closer physical limits may be obtained:—

1st—Closer chemical control on raw material

2nd—Addition of alloying elements.
3rd—More uniform heat treatment.

The third item has been solved by the furnace manufacturers; the other two can be solved if the consumers are willing to pay the extra cost.

Written Discussion: By R. L. Kenyon, supervising research engineer, American Rolling Mill Co., Middletown, Ohio.

This paper by Mr. Pulsifer shows the appreciation which the author has for the value of statistical methods in the analysis of physical test data. As Mr. Pulsifer says, this method has long been used in insurance, biology and similar sciences but it is rather recently that the engineering world has begun to recognize the possibilities of applying this branch of mathematics to the solution of some of its most important problems. Among these might be mentioned: selection of proper size of sample, study of the variation in a material as a preliminary study to writing specifications, significance of the averages of samples of various sizes (usually small), elimination of variations in a material which should not be left to chance.

It is gratifying indeed to find a paper such as this one in which such a large amount of data is given on the variation of a product because just this information should be in the hands of those whose function it is to write specifications. Too many specifications set limits without any foreknowledge of the natural limits imposed by economic and operating conditions. Data such as these should be very helpful to engineers who may have occasion to write bolt and cap screw specifications. On the other hand, if properly treated, the same data should be of immense value to the bolt manufacturer in helping him analyze the causes for the variation found and eliminate them.

The purpose of these comments is to emphasize the importance of publishing such an analysis of data as contained in this paper and to suggest a few additional treatments of such data to help answer some of the questions indicated above.

These treatments are not original with the writer but are so useful that it is thought worth while to call attention to them in connection with this paper by Mr. Pulsifer. The treatments are based on standard statistical functions which are defined in all standard tests. The theory has been specifically adapted to such problems as these by Dr. Shewhart of the Bell Telephone Laboratories.

It is suggested that the standard deviation rather than average deviation be computed as the latter cannot be used in any further computations. The standard deviation is almost as easy to calculate and can be used in testing the data further by numerous other legitimate computations. If the standard deviation is known, the simple test of "average plus or minus three times the standard deviation" can be applied to see whether the values came from a "constant system of chance causes." This has been computed from the values taken from the distribution curve of sclerescope tests in Fig. 1 of the paper and Brinell Hardness tests in Figs. 6 and 7. In all of these cases, there are values outside of the limits ($x \pm 3$) which indicates assignable causes for variation. If the original data were available, the records of these exceptional tests might furnish an explanation of these causes.

If the data can be divided into rational sub-groups, the averages and standard deviations of these sub-groups could be studied. This furnishes further tests for presence of assignable causes of variation. The data as presented in the paper are not in such a form that this kind of an analysis can be made although the original data may possibly be re-arranged so as to make this possible.

If we can satisfy ourselves that a given distribution is approximately normal, we can then calculate the size of sample (or number of tests) required so that their average will be within a given amount of the true average a certain proportion of the time. For example, the distribution of Scleroscope hits (Fig. 1) approaches that of the normal probability curve. If it is desirable to know how many Scleroscope hits to take in order to determine the average Scleroscope reading on this 0.750 inch diameter bright drawn wire within, let us say, two points of the true average hardness 99 times out of 100, we find the answer to be five. But in order to have an average within one point 99 times out of 100, 20 readings would be required. These values have been taken from a chart prepared by the writer and included in a paper "A Study of The Errors of Averages," presented last June before the A. S. T. M. at Atlantic City by Messrs. Humes, Passano and Hayes.

This discussion is offered not as a criticism of the original paper but rather to help stimulate interest in data of this kind, and to indicate a few additional treatments to which it may be subjected which may yield useful results.

Written Discussion: By H. B. Hanson, engineering laboratory, Ford Motor Co., Dearborn, Mich.

The impression one receives on reading this paper is that the term "Fine Bolts" is a misnomer as applied to a product having such a broad range as 60 points Brinell or more and the correspondingly broad ultimate tensile and yield points. Obviously if all of such production is included in the term "fine bolts" the word "fine" ceases to be a qualifying adjective.

We agree that all true tests should be included in a paper of this nature but strongly disagree with the author's attitude in including tests on Brinell impressions "made too near the edge" and the inclusion of these is evidently reflected in the curve illustration for 0.12 per cent carbon steel wire, and consequently the tests cannot be typical of distribution curves obtainable on commercial steel bar even of such broad tolerances as S.A.E. specifications.

It would appear that at least part of the "spread" of the curves may be due to inherent testing machine errors.

Many of the author's curves for example in Figs. 1, 3, 4, 6, 8, 9, 10, 12 show data points on the zero of the ordinate axis.

We would take exception to the author's expression "The material used for the cap screws tested in Fig. 2 was the same as that used for the tests of Fig. 1 except its smaller size."

To have said "of similar composition" would have been more logical unless the author intends to convey the idea that the stock was rolled or drawn down to the $\frac{7}{8}$ inch diameter.

The inclusion of complete tests taken on the stock as received would have greatly enhanced the value of the paper.

The existence of any bolts of zero elongation as shown in Fig. 7 indicates a very mediocre condition.

We cannot agree with the author's contention that the strengthening effect of the threads is so marked. We believe that a more adequate explanation and certainly a more feasible one lies in the error of assuming the root diameter to be the basis for calculating the ultimate tensile strength and yield point. There is undoubtedly a marked strengthening effect due to cold working such as rolling the threads but we have not been able to detect an appreciable work hardening effect on cut threads.

The decrease in elongation is readily explained by the effect of stress concentration. Both these points are corroborated by the decreased effect of the threading on the larger diameters.

On the twenty-second page we notice "Between the sizes of ¼ and ¾ inches, fine threads the full stem area is only from 51 to 26 per cent greater than the root areas." This must apply to bars of the major diameter and having cut threads or threads rolled on a portion extruded to the pitch diameter, but rolled threads more usually use a bar of P D or less.

We would like to have seen a series of tests on wire stock as received and as treated using a test section consisting of a bar having a cut in one series and rolled in another series around the circumference of the stock, the V having the same physical size and relation to the stock as a standard thread. We believe the comparison of these tests with the stock and with the finished bolts would have been illuminating.

We agree that better combinations of properties are available if necessary and it only requires the specification of such on the part of the user, to make these bolts commercially available.

Written Discussion: By J. L. McCloud, engineering laboratory, Ford Motor Co., Dearborn, Mich.

This is an interesting way of demonstrating what is common experience in mass production, spread of values.

I should like however to take exception to the indication that the rolling or milling of the threads is responsible for the high tensile values reported. The author shows in his micrograph Fig. 13 how rolling of the threads has compacted the grain size and changed the direction of the usual lines. He has also shown in his table on the eighteenth page how rolled threads may be 12 per cent stronger than cut threads. But I believe that he has passed over rather lightly the "necessity of fracturing across one or more thread crests." I believe that this is more important than "strengthening effect of greatly shortened test sections." Consider a screw thread as a cylinder, whose diameter is the root dimension, with a surrounding helical balcony. See Fig. 1.

It is of course well realized that the minimum cross sections is one normal to the cylinder axis, so it is also true with this modified cylinder. Examining this modified cylinder (screw threads) it is at once apparent that any normal cross section will be greater than the root cylinder section.

If too we consider the dimensions of standard screws I believe we can explain why, as noted on the twenty-fourth page of the paper, the larger the bolt the less the straightening effect of the threads.

In a ¼-inch 20-screw thread the area of a cylinder of the major diameter is 0.0491 and of the root diameter cylinder is 0.027, that is 45 per cent less. In a ½-inch 13-screw thread the area of a cylinder of the major diameter is 0.196 and of the root diameter cylinder is 0.126, that is 35.6 per cent less. In a ¾-inch 10-screw thread the area of a cylinder of the major diameter is 0.442 and of the root diameter cylinder is 0.302, that is 31.6 per cent less. These figures would no doubt be better demonstrated if the values in Table II and III were expressed in the form of frequency curves with such variables as types, steel and treatment were eliminated.

In this same condition I believe the author has in mind bolts whose main stem is about pitch diameter size in the statement on the nineteenth page rela

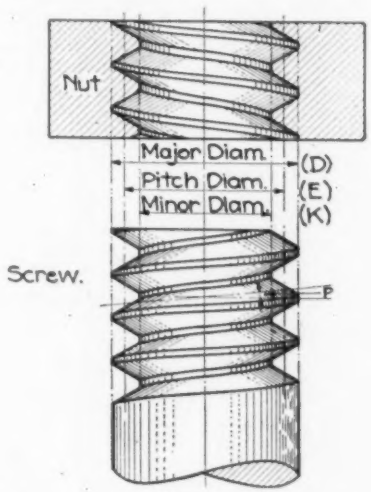


Fig. 1

tive to bolts failing in the main stem section. This is true of certain cold headed rolled thread screws but by no means all, nor is it at all customary for milled from the bar bolts.

I should also like to ask if on the eighteenth page the (20) in the table refers to threads per inch? And if that is so if some other size than $\frac{7}{16}$ inches was indicated in the coarse threads?

In the author's summary I believe that a more appropriate expression than "finer properties" would be superior combination of properties. But the statements next indicate that such finer properties are not yet obtainable, which is regrettable in bolts. I should agree that Brinell is for bolts ⅜ inches and over a most satisfactory control test and 50 pounds ample. On the other hand for pieces used in tension why not check directly by tension specifications which are quite easy to carry out when one's test pieces are all prepared.

We are glad that bolt manufacturers will supply "finer bolts" and no doubt specifications will be accordingly altered.

Written Discussion: By H. P. Coats, research chemist, Firestone Tire and Rubber Co., Akron, Ohio.

The author has given us the physical properties of a dozen different classes of bolts. He has illustrated these properties, not by a couple of samples in each case, but by one hundred to five hundred samples. It is a decided advantage

to have graphs of a large number of samples. We are then able to estimate between what limits a certain class of steel will vary.

The author's work has been done on the finished product. He has shown that the deviation of yield and tensile is greater on the finished product than on a more simple shape. This fact probably applies to other commercial products. Therefore, the engineer is again exonerated for using a "large factor of safety."

It is, of course, not to be expected that the frequency curves for the physical properties of a series of bolts will present the typical bell shaped curve illustrated in Fig. 1 for scleroscope hardness on a single sample. The reasons for variation are mentioned by the author as starting with the manufacture of the steel and extending to the heat treatment and cold work on the finished product. Probably the high sulphur stock illustrated in Fig. 12 shows the greatest range of variation. The dispersion from any average yield-point is especially noticeable here. One wonders if the heterogeneity caused by the manganese sulphide inclusions does not contribute somewhat to this wide variation.

The author says "when finer bolts are needed, they are pretty sure to be supplied." This statement is interesting in view of the fact that the curves for yield point and tensile strength have a skewness ("tail") toward the higher value. It is not probable that this is an accidental dispersion. Nor is it probable that some personal error can account for it. Would it not be advisable to investigate this skewness toward greater strength as a possible means of discovering how to make finer bolts?

It seems to me that when we are ready to make a steel more homogeneous microscopically; that is, with fewer nonmetallic inclusions, we will have finished products of steel with much less variation.

It would have been easier to follow the curves if the author had employed the same cross-section for plotting all the curves. If the data had accompanied the curves, it would have assisted me, though, of course, space must be preserved.

Written Discussion: By O. V. Greene, metallurgical department, Carpenter Steel Co., Reading, Pa.

The accumulated data of many investigations is often incorrectly analyzed and even discarded altogether because of hopeless inconsistencies. The statistical method, however, yields results from which very useful conclusions can be drawn. H. B. Pulsifer has successfully applied this large number research to a group of problems which probably could not be satisfactorily solved by ordinary methods. The contribution should therefore be welcomed by bolt manufacturers and consumers alike.

The law of probability portrays actual experience rather than theoretical considerations. It is consequently of considerable practical advantage in determining causes and particularly remedies for unsatisfactory results. In other words, such a method should be an excellent barometer of quality. It should aid in the selection of raw materials and treatments for certain desired results, and in the development of new processes. The relation between decrease in "spread" allowed by more rigid specifications and price, is at once also available by this method of analysis.

A point in the paper that cannot be stressed too much, is the relation be-

tween standard tensile specimens and the finished threaded bolt. Too often there is a disagreement between manufacturer and purchaser when the results of the tests of threaded sections do not agree with the expected properties or specifications based on smooth turned bars.

C. T. PATTERSON:[1] In presenting the data as a probability curve, do not feel too badly about the spread of that curve because that includes also the probability of errors in the testing apparatus and methods. If we had a material which was absolutely uniform and tested it a large number of times by the Brinell or scleroscope, we would get a bell-shaped curve.

H. F. MOORE:[2] There is one point which I think was not emphasized sufficiently, and that is the difference between the effect produced by cold rolling and the effect produced by cutting. You will remember that when the micrographs were thrown on the screen the statement was made that the cutting did not affect the crystalline structure as far as the general crystalline arrangement was concerned. I think you will see that the cutting does make tears at the root of the thread while the cold rolling process changes the crystalline structure at the root of the thread and has less tendency to make tears than the cutting process. I will add that at the University of Illinois we have found that not only are the *static* properties of materials improved by suitable cold rolling but also there is a considerable improvement in the *fatigue* properties. We have found the fatigue strength of bolts are a very troublesome practical problem. I would refer those who are interested to the work recently done in Germany which shows that any kind of a notch made with a blunt tool which cold works the metal does less damage than the same size and shape of notch made with a sharp cutting tool. I would refer you to a summary of this work and an article by Mr. Heydecamp which appears in *Iron Age* for Sept. 18 and Oct. 2, 1930.

SAMUEL HERSCH:[3] I can check up on Prof. Moore's statement by stating that in thread stripping tests, previous to heat treatment, roll threaded bolts show an increase in strength over that of cut thread bolts approximately 13 per cent.

The tests that Mr. Pulsifer made were all very fine, but when bolts are being turned out on a production basis, and hundreds or thousands of bundles of wire are being cut up to make these bolts, the resulting physical properties due to chemical analysis only vary considerably. At our laboratory we made tests for the carbon content on approximately 4000 bundles of wire, and we found that there was a variation from 0.20 to 0.46 per cent carbon in some of the heats. Due to rigid inspection at the steel mill this condition has been eliminated to a very satisfactory degree. However, heat treat bolts made from wire of this varying carbon content, and try to get them within Brinell hardness limits of 196-241 or 207-255. It cannot be done. You must start out with wire having a uniform chemical analysis.

If bolts were made from ordinary "run of mine" wire as shown above, and heat treated as 0.30-0.37 per cent carbon steel, we can assume that approximately

[1]Metallurgist, Solvay Process Co., Syracuse, N. Y.

[2]Professor of engineering materials, University of Illinois, Urbana, Ill.

[3]Metallurgist, Superior Screw and Bolt Mfg. Co., Cleveland.

18 per cent of the bolts would be out of the Brinell hardness range of 196-241. If the bolts were heat treated to obtain a Brinell hardness of 196-241 on a 0.30 to 0.40 per cent carbon steel, then we can assume that approximately 6 per cent of the bolts would be outside the above hardness limits.

There are many factors which must be taken into consideration in the manufacture of high quality bolts, two of the most important ones being the analysis of the raw material (both physical and chemical) and the heat treating equipment together with the part which the human element plays.

To heat treat 150 or 500 bolts by themselves in a continuous furnace, which normally turns out from 800 to 1000 pounds of bolts per hour, is very difficult, and the results are not satisfactory.

R. E. CHRISTIN:[4] I did not quite understand the author's meaning when he said *fine* bolts. We consider all bolts made for the automobile manufacturers as being *fine* bolts. This paper should be an aid toward bringing the specification writer and manufacturer together. When the automobile manufacturer's specification writer makes up the specifications, I do not believe he has in mind the fellow on the other side of the fence. He makes up the specifications calling for a Brinell range of approximately 40 points in a carbon range of approximately 10 points. We have found one heat, especially on a carbon steel, covering the entire Brinell range. The carbon probably will not vary more than 4 points. If you let a lot of that Brinell limit go over in a particular heat, say 10 points, you get a comeback from the automobile manufacturers. The bolts are not uniform. So all you can do is take the bolts back and process them in small batches in order to get within the manufacturers' limits. Therefore I would suggest that the manufacturer's specification writer would either limit his carbon range or increase his Brinell range when he is talking about carbon bolts. We have found on carbon steel specifications a range of 40 points Brinell and the same range given on nickel steel bolt specifications. We can get it on nickel steel. We can get within 20 or 30 points Brinell, but they give us 40 points, and we enjoy that. But when it comes to carbon steel Brinell ranges, we would appreciate a little wider range. If you take a carriage bolt which has a thin head and a cap screw, you will find there will be some difference between the Brinell on the head and on the body. Especially is this true when the bolt is made from hot-rolled material against cold drawn material. In cold drawn wire the difference in hardness between the head and the body is not so great, but on hot-rolled material where you are subject to decarburization, there is some difference between the hardness on the shank and head. But the manufacturer and consumer seem to be satisfied with taking the hardness on the head. When you begin to test those bolts for physical properties, you will find quite a considerable spread for the Brinell hardness that you have taken on the head. It seems to be an error to tie up the physical properties on the bolt when you have taken the Brinell hardness on the head. We also would suggest some tests to be made in determining physical properties on bolts by testing these bolts on a torsion test. If you recall on service tests that this bolt, especially in the assembly, is subject to torsion as well as to tension, but when you pull it in tension the bolt never breaks like a tensile piece. That might be

[4]Metallurgist, Columbus Bolt Works Co., Columbus, Ohio.

some explanation for Mr. Pulsifer's findings on the erratic results he obtains on testing threaded material against smooth tensile pieces.

I have found Mr. Pulsifer's paper very interesting. He has covered a considerable range, and I would like to say that we desire very much to have the manufacturer and consumer get hold of this paper. I believe there is a lot of good food for thought.

I would like to suggest to the author the use of Rockwell "B" hardness on cold drawn wire and bolts in place of the scleroscope hardness, as a surer means of detecting differences due to cold drawing, heat treating, and one or more reductions. It has been our experience that the surface hardness together with the "internal" hardness are factors to be considered in checking physical properties of bolt wire and bolts before and after heat treatment, such as low and high temperature annealing.

E. S. TAYLERSON:[5] My knowledge is very limited in regard to the manufacture and testing of bolts. Dalby[6] studied the strength of bolts many years ago and found that the actual shape and fit of the thread is even more important than the quality of material. It has already been brought out in the discussion that, if the data has been plotted in percentage of the total number of tests the different diagrams would have been more comparable. I am in thorough agreement with that statement, but would carry it a little farther by also plotting the percentage deviation of the particular quality measured. This deviation can be measured from the average, or the optimum or the specification value. The area under the curve would have then been the same for all examples and they could be compared at a glance. Some years ago in a discussion of a paper by Dr. Saklatwalla and Mr. Chandler[7] I advocated the use of specially ruled probability paper for such curves. The probability curve if normal, is graphically reduced to a straight line. While the mathematicians who deal in statistics of probability consider this to be rather an unscientific method, I regard it as a very valuable method especially for work such as physical testing that is not of very high accuracy.

Author's Closure

The author is well satisfied with the appreciative remarks by Mr. Harvey. In fact, the final comments of his contribution are not of a nature to disturb the kindly impression at first created.

Since Mr. Harvey is not satisfied with the apparent status of the straight carbon bolts as given in the distribution curves he selects two coils of wire, manufactures the bolts and then tests certain "groups," some before and some after treatment. We are unable to attach much weight to the limits disclosed by these tests because Mr. Harvey fails to state the number tested. He indicates no distribution of the results.

But, in the opinion of Mr. Harvey, his results have restored the justice

[5]Engineer of tests, American Sheet and Tin Plate Co., Pittsburgh.

[6]Strength and Structure of Steel. W. E. Dalby. Longmans.

[7]B. D. Saklatwalla and H. T. Chandler, "Application of the Mathematics of Probability to Experimental Data as a Basis for Appropriate Choice of Ferrous Materials, TRANSACTIONS, A. S. S. T., Vol. 10, Page 205.

due the straight carbon bolts and established the assumption that continuous travelling hearth type furnaces make more uniform product.

Others have been hopeful of seeing even more substantial evidence of the superiority of continuous furnaces.

A short while ago the author undertook a pilot test for the very purpose in question. A small lot of 0.40 per cent carbon cap screws from one coil of wire was heated for quenching in one of the recent installations of continuous furnace. The cap screws were Brinelled after the quench and then drawn

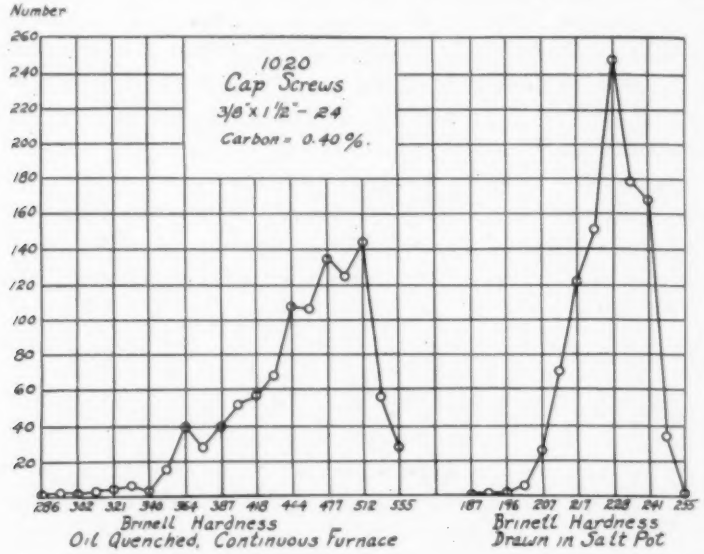


Fig. 1. Distribution Curves Showing Brinell Hardness of 0.40 Per Cent Carbon Bolts after quenching from Continuous Furnace and after Tempering in Salt Pot Furnace. The Lot Contained 1020 Bolts.

to the customary range of Brinell 207-255. They were then again Brinelled. The lot was found to contain 1020 cap screws.

The results are plotted on the accompanying diagram to show the distribution of the results. The second curve shows that only eleven pieces were found to be outside the 207-255 range.

But this is neither better nor worse than similar tests made by quenching from the rotary gas-fired retorts as indicated in the original paper.

Further evidence will, of course, be eagerly awaited by all bolt manufacturers.

The title of "Fine Bolts" refers largely to the physical dimensions and distinguishes those considered fine hot headed bolts, bolts with reduced stems and whole classes of bolts made with dimension tolerances greater than a few thousandths of an inch. If Mr. Hanson still insists that these cap screws and bolts are not "fine" because of property spread, we shall probably have to omit the adjective.

Mr. Hanson is correct about the data points on zero of the indicated scales; there were no results found with those values. They were necessarily indicated when other specimens had more extreme values.

In referring to "material, was the same" on the seventh page, the author intended to convey the meaning "of similar composition, grain size, and physical properties."

As to the zero elongation of the high manganese bolts, we shall have to concede a point to Mr. Hanson. Elongations were called zero when less than 1/64 inch, actual. The measuring of minute elongation on a fractured bolt is difficult. Further, however, we may state that our experience has been that the high manganese steels have decidedly less elongation than other steels at the same hardness. We do not check other investigators who find them as ductile as many straight carbon or many alloy steels.

The views of Mr. Hanson on the cold working by rolling threads and the lack of it with cut threads is in agreement with the author's data and helps answer Mr. McCloud's remarks.

Mr. Hanson states, "rolled threads more usually use a bar of P. D., or less." In our plant it is common practice to "mill" or "extrude in the die" full sized wire for roll threading. I do not understand how a full sized thread could be rolled on a stem of less than the pitch diameter. It is not done in our plant. Bolts with threads the full length of the stem are, of course, commonly roll-threaded from P. D. wire.

Mr. Hanson evidently checks Mr. McCloud about the matter of getting better bolts by merely specifying them. The author considers one of the greatest improvements toward better bolts will be the use of better and more uniform steel. This will likely demand electric furnaces instead of basic open-hearth steel, and will cost more. If better steel is made in open-hearth furnaces, as is not improbable, it again points to more expensive steel. If the better bolts are needed they can be "specified" with this in mind.

Replying to Mr. McCloud's exception that rolling threads increases the tensile strength of the material or causes greater strength than cut threads, the figures on the eighteenth page furnish adequate answer. These figures are typical of the common result and have been checked repeatedly on many materials.

Cut threading gives no appreciable distortion or cold working of the material. This is easily observed by the microscopical examination of etched sections taken from cut threaded bolts. Our laboratory has prints of many such sections.

Roll threading unavoidably cold works the material, raising the yield point and tensile strength and decreasing the elongation. This method of manufacture affords a very practical way of avoiding heat treatment and yet providing a bolt with superior physical properties. It is especially applicable to low carbon ferrous alloys and nonferrous alloys that are not easily improved by customary heat treatments.

Although roll threading has been practiced by our company for some twenty years it is only recently that we have had figures to demonstrate its advantages on the physical properties of materials.

Regarding the statement of Mr. McCloud about bolts failing in the stem, the statements in the original paper refer to bolts of full stem size. No bolts of less than full stem size were considered in the paper. Bolts with stems less than full size and roll threaded might well be expected to fail in the stem.

The numbers in parenthesis in the table on the eighteenth page indicate the number of pieces pulled for the average figures given.

The inference about manufacturers supplying better bolts on specification might well be expanded to include the kindly offices of the purchasing agent.

Mr. H. P. Coats evidently has sympathy for the shortcomings of the paper for he mostly answers his own queries.

However, he does touch a very tender and vital issue about making *all* of the bolts like the best. It is not infrequently rather embarrassing to attempt an explanation of why all the bolts are not like the best.

The use of more uniform and perfect steel is obviously a prime step toward manufacturing bolts with better properties and less spread. Commercial basic open-hearth steel as made into bolt wire is very far from perfectly clean and uniform material. Systematic microscopical inspection reveals surprising ranges in structural constitution and many types of interesting but excessively annoying "structural diseases."

It may also be surmised that future studies will disclose causes for accelerated and delayed transformations and coalescences. The conditions for producing uniform structures will then be made use of and far less "spread" occur in the final product.

Even when all of this has been done there remains the probability that there will be tails to the distribution curves: worst, average, and best bolts.

The remarks of Mr. Greene in appreciation of the author's efforts are very welcome. One can easily believe that Mr. Greene is an easy convert to the advantages of probability studies.

Incidentally, Mr. Greene does not fail to notice that in the making of better bolts the accomplishment may be worth the cost. That is, he realizes that they must be purchased as well as specified.

COLD HEADING DIE LIFE

By A. S. Jameson

Abstract

The development of cold heading in the bolt industry has presented numerous die problems. This paper describes an investigation carried out for the purpose of obtaining the maximum die life from a certain type of cold headed bolt die.

The maximum production of a cold heading die is dependent on five main factors, namely: die steel, die design, die heat treatment, bolt material, and machine operation. Though die steel and heat treatment are primarily dealt with, each of the other factors is discussed and their relation to die life is pointed out.

Special stress is laid upon the important part played by the depth of the hardened case of the die produced by heat treatment on the die life.

An effort is made to stimulate interest in the problems of cold heading die life by the development of a series of questions in connection with the investigation.

COLD forming is replacing hot forming as a means of shaping steel. The rapid growth of the cold forming industry has produced a metallurgical problem of great magnitude—the problem of the production of dies capable of withstanding the repeated impacts of a relatively rigid body. The fact that cold forming is gaining in favor as a means of shaping steel is indicative that considerable advances have been made toward solving this problem, for the life of the cold forming dies must be of certain length in order that there be an economic advantage in the cold forming process.

As the whole field of cold forming is too extensive to deal with in a single article, this paper is confined to the solution of a specific die problem. The limiting of the field does not detract from the value of the information obtained as all of the problems of die life are interrelated. The problem in this instance is to

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, A. S. Jameson, a member of the society, is metallurgist, West Pullman Works, International Harvester Co., Chicago. Manuscript received May 28, 1930.

obtain the maximum production from a die used to cold form or cold head a type of bolt shown in Fig. 1. Specific data is given on the relation of die life to steel and heat treatment. The bolt is used extensively in the farm equipment industry and is known as a plow bolt.

Obtaining the maximum production from a cold heading die is dependent on five main factors, steel, design, heat treatment, bolt material, and machine operation. It is convenient to divide the material into two parts, the first will deal with the die steel and the second with the dies.

Part I

Die Steel

Chemical Composition

It is generally conceded in the bolt industry that plain carbon or carbon steels containing small percentages of vanadium give the most satisfactory results for cold heading dies. Four steels were selected for the investigation and are designated as A, B, C, and D. They were plain carbon steels of the analysis shown in Table I.

Table I
Chemical Composition of the Steels in Per Cent

Steel	Carbon	Manganese	Sulphur	Phosphorus	Silicon	Chromium
A	0.96	0.28	0.014	0.020	0.22	0.03
B	0.96	0.28	0.021	0.013	0.20	0.07
C	1.10	0.30	0.012	0.014	0.15	0.05
D	1.00	0.45	0.012	0.024	0.22	0.12

Steel A is the product of an open-hearth furnace, steel B that of a crucible furnace and steel C a higher carbon melt from a crucible furnace. These steels are of the usual chemical composition. The composition of steel D is interesting for it is of higher manganese content than is found in carbon tool steels used for cold heading work. The current metallurgical opinion is not in favor of the use of manganese content higher than 0.30 per cent in high carbon steel dies for cold heading. The chromium content of 0.10 per cent in steel D approaches the range where chromium will modify the hardening properties of carbon tool steels. The

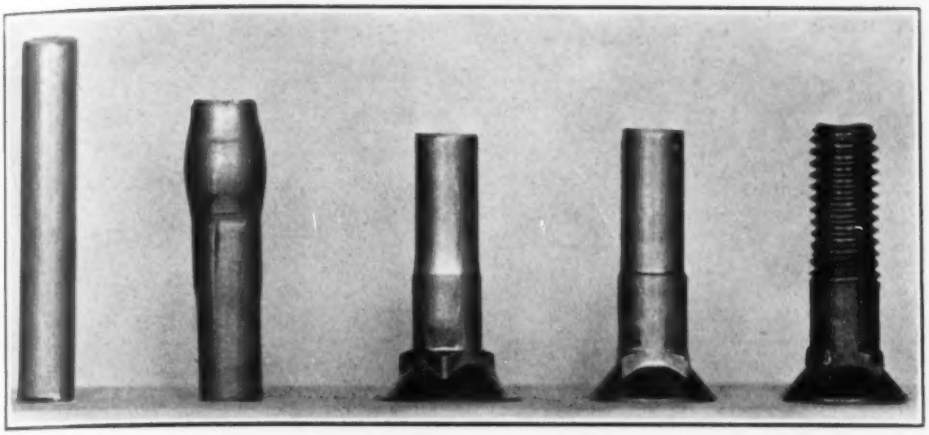


Fig. 1—Plow Bolt and the Sequence of Manufacturing Operations. Left to Right: Wire Length, First Upset, Blank, Trimmed Blank, and Finished Bolt.

steels were obtained from the mills in 3⅝-inch round bars in the annealed condition.

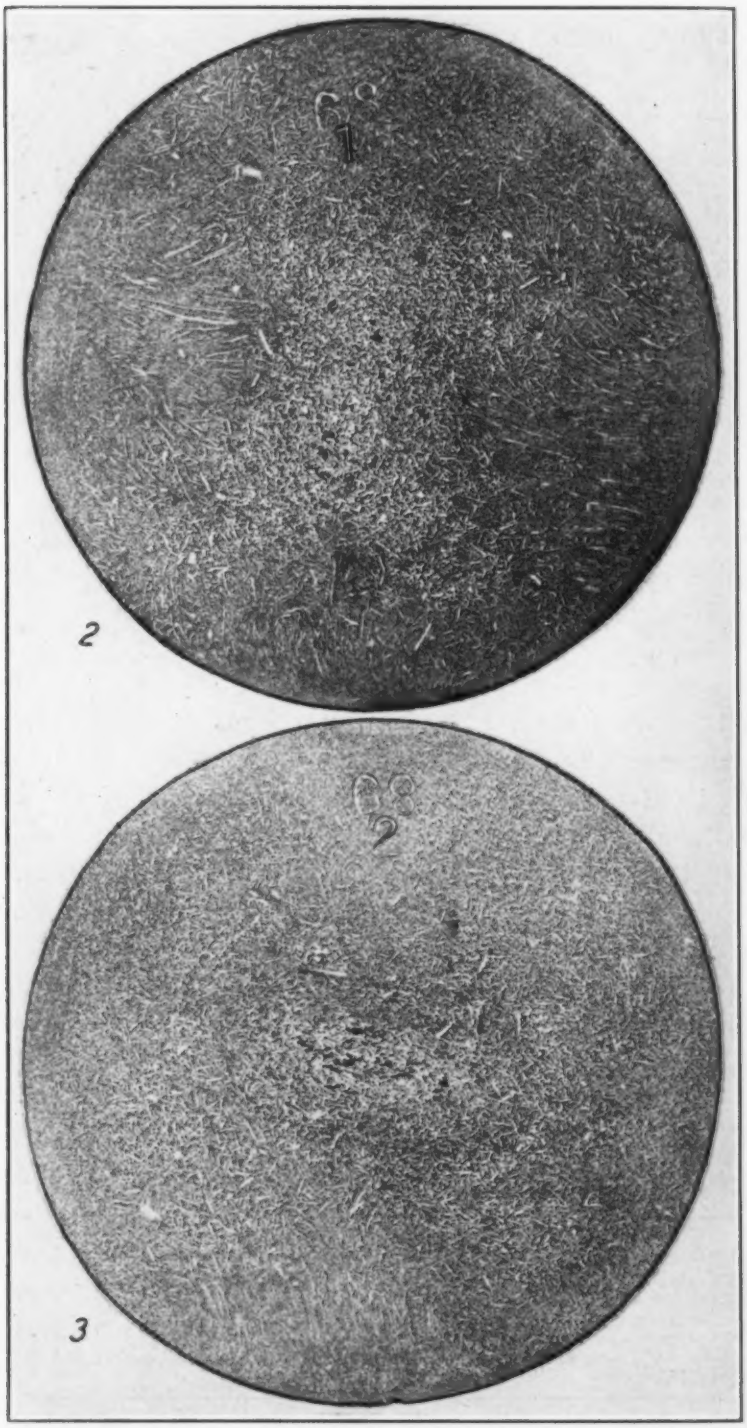
Inspection of Steel Bars

The bars were subjected to the usual inspection by deep etching, microscopic examination and hardness tests. Deep etching was carried out on ½-inch disks, from both ends of the bars, by emersing the disks heated to 200 degrees Fahr. in a boiling 1:1 solution of hydrochloric acid for a period of ten minutes. Figs. 2 and 3 are representative of all the bars, and are macrographs of the ends of the same bar. By our laboratory standards on tool steel for cold heading these bars would be considered of fair quality entirely suitable for use. The annealed structures are described in Table II and the descriptions illustrated in Figs. 4, 5, 6, and 7.

Table II
Annealed Structures of Steels

Steel	Description	Brinell Hardness
A	Carbides 75% spheroidized, remainder lamellar pearlite, Grain open.	207–217
B	Carbides 95% spheroidized, some large particles of carbide, remainder lamellar pearlite. Grain open.	149–156
C	Carbides completely spheroidized some large irregular carbide particles. Grain close.	143–156
D	Carbides 50% spheroidized, remainder pearlite and sorbo-pearlite. Grain close.	170–179

The large carbide particles, probably remains of the network structure, are not particularly desirable, the hardness values are within a range which we find to have suitable machining properties.



Figs. 2 and 3—Macrostructures Representative of the Bars of Steels A, B, C, and D. Etched 10 Minutes in 1:1 Hydrochloric Acid. Photographed actual size, Slightly Reduced in Printing.

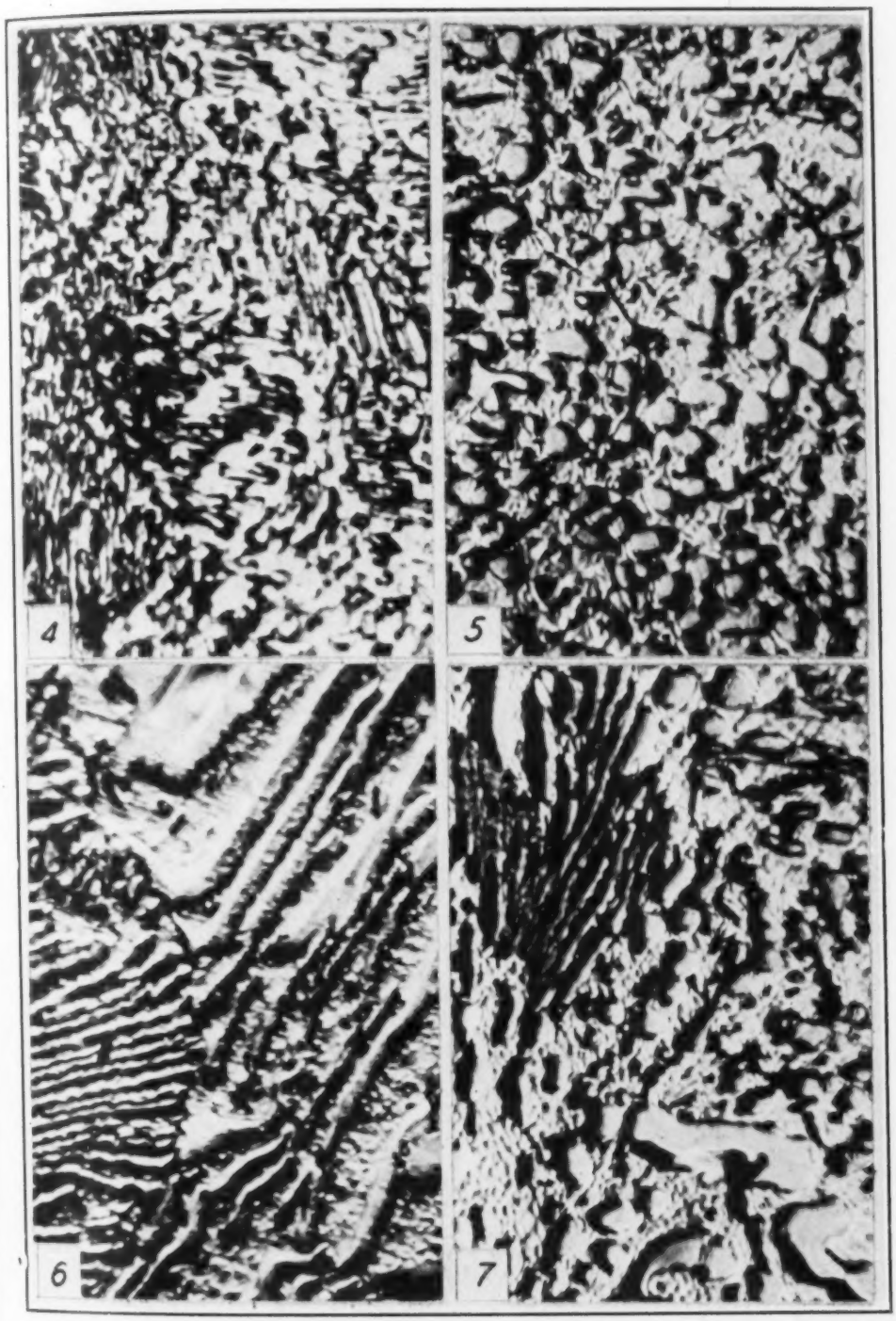


Fig. 4—Photomicrograph of Sorbo-Pearlite Structure in Annealed Steel. × 2000. Fig. 5—Photomicrograph of Spheroidal Cementite in Matrix of Ferrite in Annealed Steel. × 2000. Fig. 6—Photomicrograph of Lamellar Pearlite in Annealed Steel. × 2000. Fig. 7—Photomicrograph of Spheroidal Cementite, Lamellar Pearlite, and Massive Cementite in a Matrix of Ferrite in Annealed Steel. × 2000.

Table III
Surface Hardness of the Quenched 1-Inch Rounds

Steel	Heated to 125°F above Ac_{1-2-3}, held 20 minutes and quenched in 3-4% brine (40-45°F)	Heated to 125°F above Ac_{1-2-3}, held 60 minutes and quenched in 3-4% brine (40-45°F)	Heated to 225°F above Ac_{1-2-3}, held 20 minutes and quenched in 3-4% brine (40-45°F)
A	66-66-66-66-65	65-64-64-64-65	64-64-64-64-63
	66-66-66-65-65	66-65-64-65-65	60-65-63-63-64
	66-66-66-64-66	64-65-64-65-66	64-64-63-64-63
	65-65-65-64-65	65-65-65-64-64	63-64-64-64-63
	65-65-65-66-65	65-65-64-65-64	64-64-64-63-63
Average	65.4	64.7	63.5
B	65-65-66-66-64	65-64-65-65-66	63-64-64-64-64
	64-64-65-65-66	65-65-66-66-66	64-63-63-64-64
	64-65-65-63-61	66-66-66-66-65	64-64-64-65-64
	64-65-66-65-64	64-64-65-65-66	64-64-63-64-64
	65-65-66-65-65	66-66-65-65-64	65-64-64-63-64
Average	64.7	65.3	63.9
C	66-65-64-65-60	66-65-65-65-65	63-62-63-62-64
	61-60-64-61-61	65-65-66-65-65	64-65-65-64-63
	65-64-62-65-66	66-65-65-66-65	65-64-62-64-62
	65-65-52-48-55	66-65-65-65-65	63-65-65-62-64
	59-58-52-44-60	65-64-65-65-65	60-61-64-61-60
Average	59.9	65.2	63.1
D	65-64-65-63-65	64-65-65-65-62	63-61-62-61-62
	65-65-65-63-62	64-65-64-63-64	62-61-59-62-62
	62-63-63-64-65	64-64-65-66-65	64-61-65-64-64
	64-64-65-64-65	63-64-64-65-64	64-63-64-64-65
	65-64-64-62-64	63-65-63-64-63	62-65-64-63-60
Average	64.0	64.1	62.7

Quenching Tests

The study of the characteristics of the steels was continued by quenching tests and later by McQuaid-Ehn carburizing tests. The quenching tests were carried out on 1-inch rounds machined from the bars. Four rounds being obtained from the same plane in the bars and representing longitudinal sections in an area half way between the center and the outside. Two quenching temperatures of 125 and 225 degrees Fahr. above the A_{3-2-1} points were used with holding times of 20 minutes and 60 minutes. Before commencing the actual heat treatment of the 1-inch rounds, critical point determinations were made. The results of these determinations are given in Fig. 8.

The 1 inch round bars were heat treated in an electrically heated and controlled furnace. They were put in the furnace at a temperature of 1200 degrees Fahr. and after equalization were heated at a rate of 5 degrees Fahr. per minute to the desired quenching temperatures. Due to the small mass of the rounds in comparison to the furnace

the A_{3-2-1} points were partially masked and their passage through this range was recorded on the temperature recorder chart in a form which is generally referred to as a hump. The actual quenching temperatures of the rounds occurred between 1510 to 1525 degrees Fahr. for the lower heat and between 1610 to 1625 degrees Fahr. for the higher heat. The rounds were quenched in a 4 per cent brine solution maintained at a temperature of from 40 to 45 degrees Fahr. The surface hardness of the quenched rounds is given in Table III.

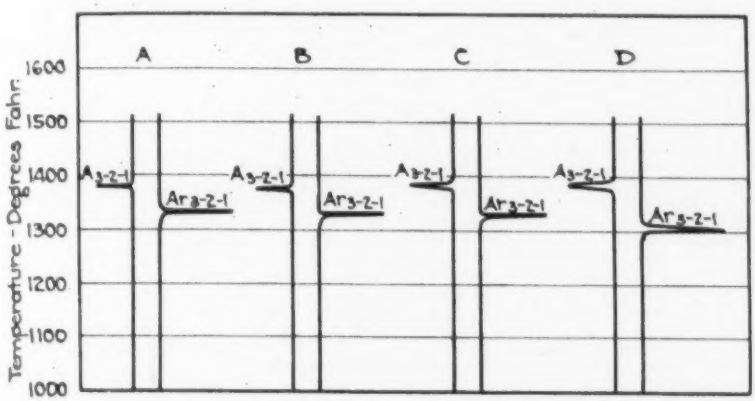


Fig. 8—Graphical Representation of the Critical Points of Steels A, B, C, and D.

The quenched bars were nicked on a flexible grinding wheel and broken for observation of the fractures. These are described in Table IV.

Cross-sections were cut through the quenched rounds in order to measure the depth of the hard case. The depth of the case was read off in sixty-fourths of an inch on the polished and etched cross-

Table IV
Appearance of Fracture of Quenched 1-Inch Rounds

Steel		Quenched from 125° F. above Ac_{1-2-3} held 20 minutes	Quenched from 125° F. above Ac_{1-2-3} held 60 minutes	Quenched from 225° F. above Ac_{1-2-3} held 20 minutes
A	Case	Very coarse	Very coarse	Very coarse
	Core	Very coarse	Very coarse	Very coarse
B	Case	Fairly fine	Fairly fine	Fairly fine
	Core	Coarse	Coarse	Coarse
C	Case	Fine	Fine	Fine
	Core	Coarse	Coarse	Coarse
D	Case	Fine	Fine	Fine
	Core	Fine	Fine	.....

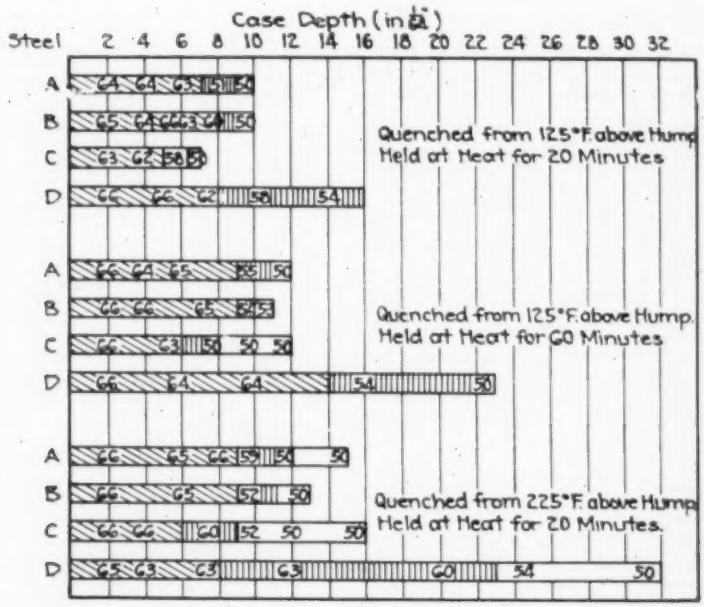


Fig. 9—Diagrammatic Representation of Case Depths of Quenched 1-Inch Rounds. The Sloping Cross-Hatching Shows the Extent of the Completely Martensitic Case, the Vertical Cross-Hatching the Martensitic and Troostitic Transition Zone and the Remaining Section Shows the Extent of the Case as Measured by Rockwell "C" Hardness Tester.

section, the depth of case being considered as equal to the extent of the martensitic structure. These measurements are supplemented by hardness readings where a reading of 50 Rockwell "C" was judged to be the limit of the case. Fig. 9 is a diagrammatic representation of the case depth. The cross-sections were also used for examinations of the structure of the quenched rounds. The heat treated structures of the martensitic cases are illustrated in Figs. 10, 11 and 12, and the cores in Figs. 13, 14 and 15.

Discussion of Quenching Tests

Surface Hardness

Generally speaking the surface hardness of the 1-inch rounds is in accordance with the findings of others, a slight falling off in the hardness of the steels is noticed when the temperature is increased in the neighborhood of 1600 degrees Fahr. The higher carbon (1.10 per cent C) steel C shows soft spots at the lower quenching temperature and short holding time emphasizing that a steel of this type should be held a considerable time at heat if a low quenching temperature is used.

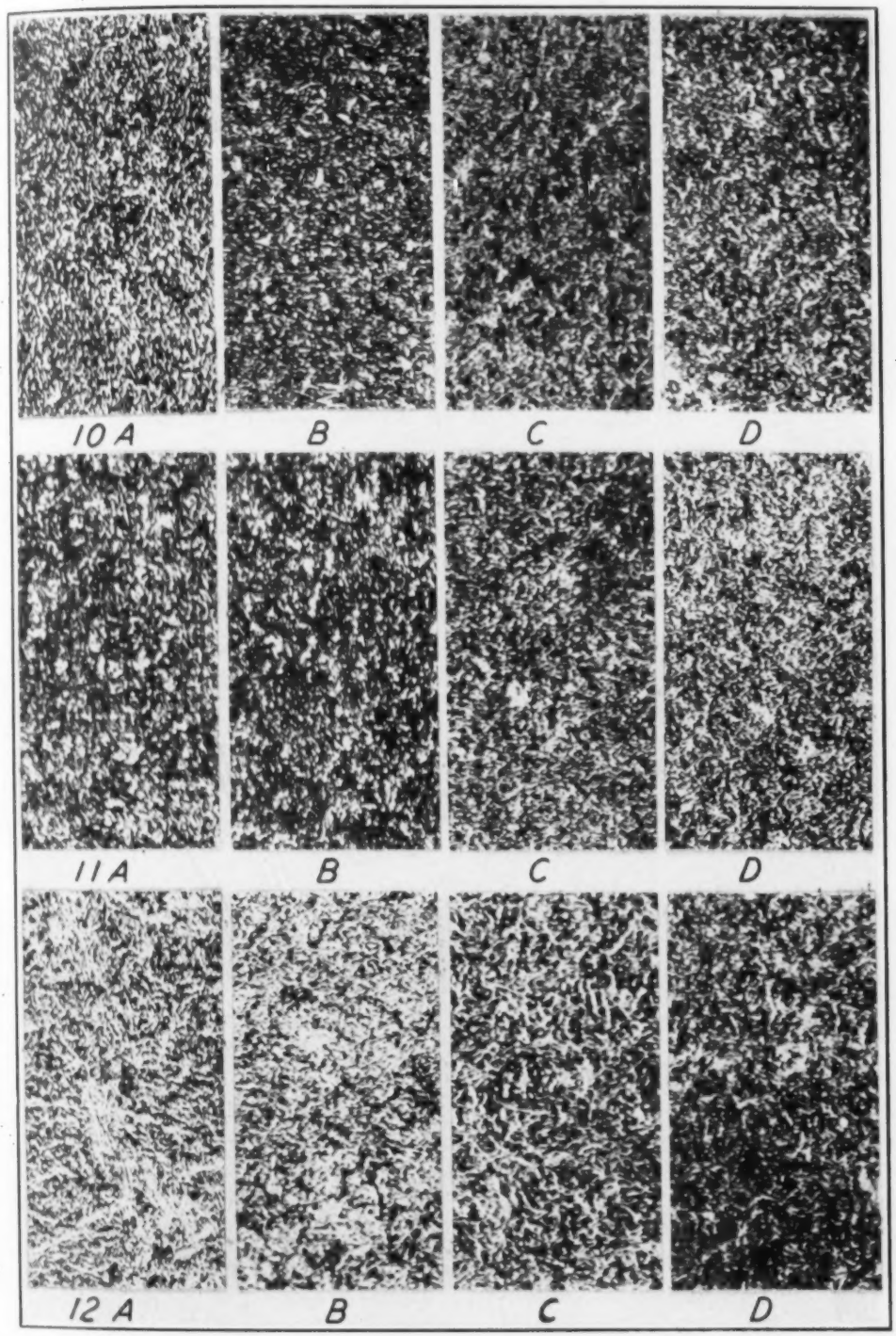


Fig. 10—Photomicrographs of Case Structures of Steels A, B, C, and D Quenched from 125 Degrees Fahr. Above Hump After Holding 20 Minutes. × 300. Fig. 11—Photomicrographs of Case Structures of Steels A, B, C, and D Quenched from 125 Degrees Fahr. Above Hump After Holding 60 Minutes. × 300. Fig. 12—Photomicrographs of Case Structures of Steels A, B, C, and D Quenched from 225 Degrees Fahr. Above Hump After Holding 20 Minutes. × 300.

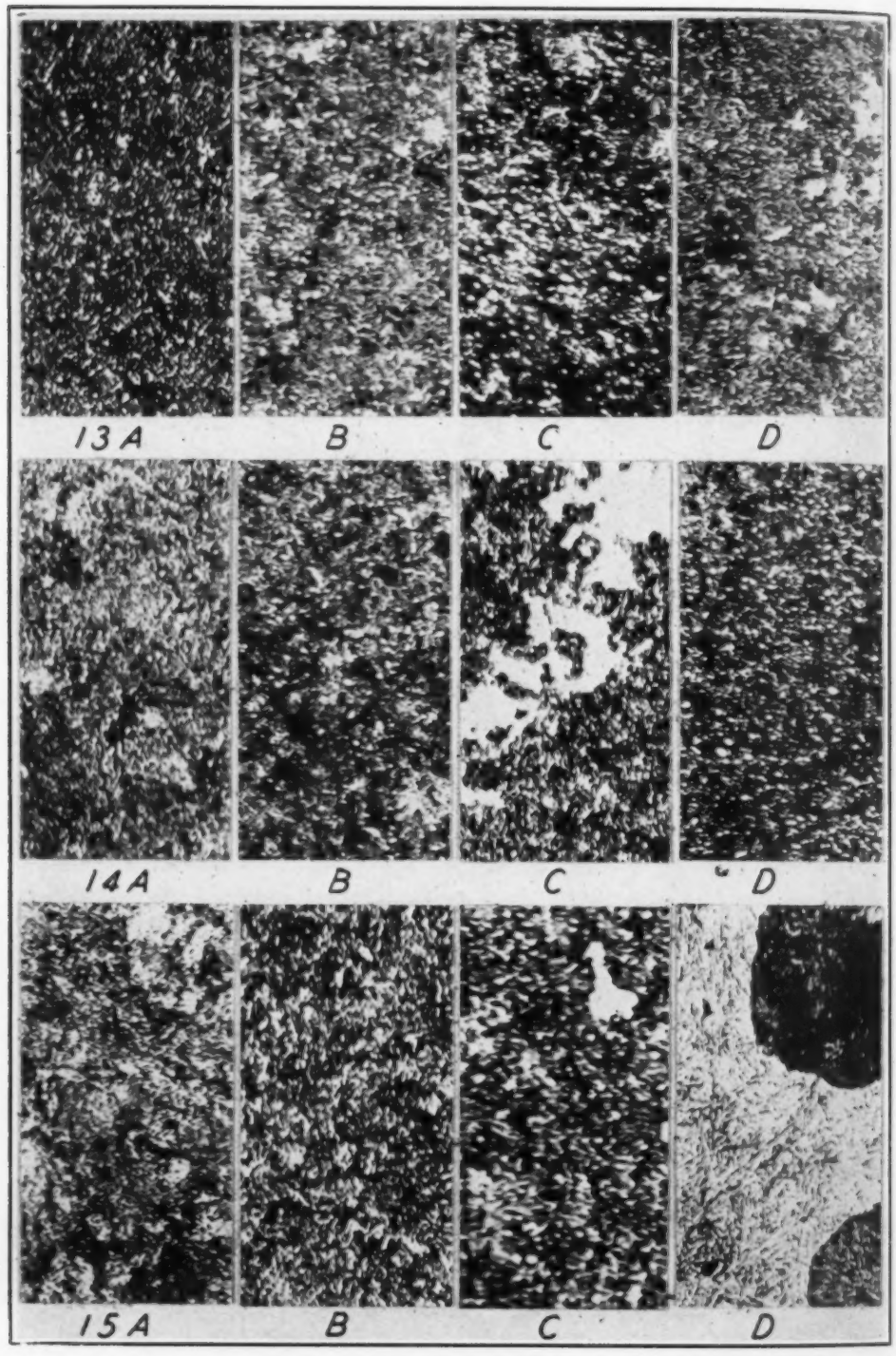


Fig. 13—Photomicrograph of Core Structure of Steels A, B, C, and D Quenched from 125 Degrees Fahr. Above Hump After Holding for 20 Minutes. × 300. Fig. 14—Photomicrograph of Core Structures of Steels A, B, C, and D Quenched from 125 Degrees Fahr. Above Hump After Holding for 60 Minutes. × 300. Fig. 15—Photomicrograph of Core Structures of Steels A, B, C, and D Quenched from 225 Degrees Fahr. Above Hump After Holding for 20 Minutes. × 300.

Fracture

The high quenching temperature produced only a slight increase in the coarseness of the fracture, that is, to the eye. Steel A had a very coarse dry fracture in both case and core. Steel B had a fracture which is representative of the usual carbon steel. Steel C had a finer case fracture than B. Steel D exhibited both a fine case and core.

The measurements of the case depth of the hardened rounds indicated that an increase in the temperature produced an increase in the case depth as did an increased holding time. By checking our observations as to the case depths, as measured by the extent of the martensitic structure by hardness tests, we reached the conclusion that the extent of the martensitic structure is not an accurate measure of the case depth. Steel C (1.10 per cent C.) consistantly showed an observed shallow case depth while the hardened case depth, as measured by Rockwell readings, equalled that of the steels A and B (0.96 per cent C.) at any rate at the higher quenching temperature. Steel D hardened to a considerably greater depth than A, B, and C. We believe this to be due to the higher manganese and chromium contents for this steel exhibited all the characteristics of a steel containing chromium and high manganese. This will be discussed later in the paper.

Microstructure

The microstructure of the cases of the quenched rounds in general indicate that the higher quenching temperatures make for increased solution and retention of the carbides. In particular more carbides are out of solution at the low quenching temperature in steels B and C than in steels A and D. The steels show an increased grain growth at the high quenching temperature more so in the case of steel "A" and less so in the case of steel D. The cores show a similar trend though to less marked extent. The soft areas in steel C after quenching at the low temperature and at the short holding time are troostitic and are associated with minimum solution of the carbides.

McQuaid-Ehn Test

The value of the McQuaid-Ehn test as applied to tool steel is not definitely proven. It is, however, agreed that it is a means of detecting variations in the physical make up of tool steels. Epstein

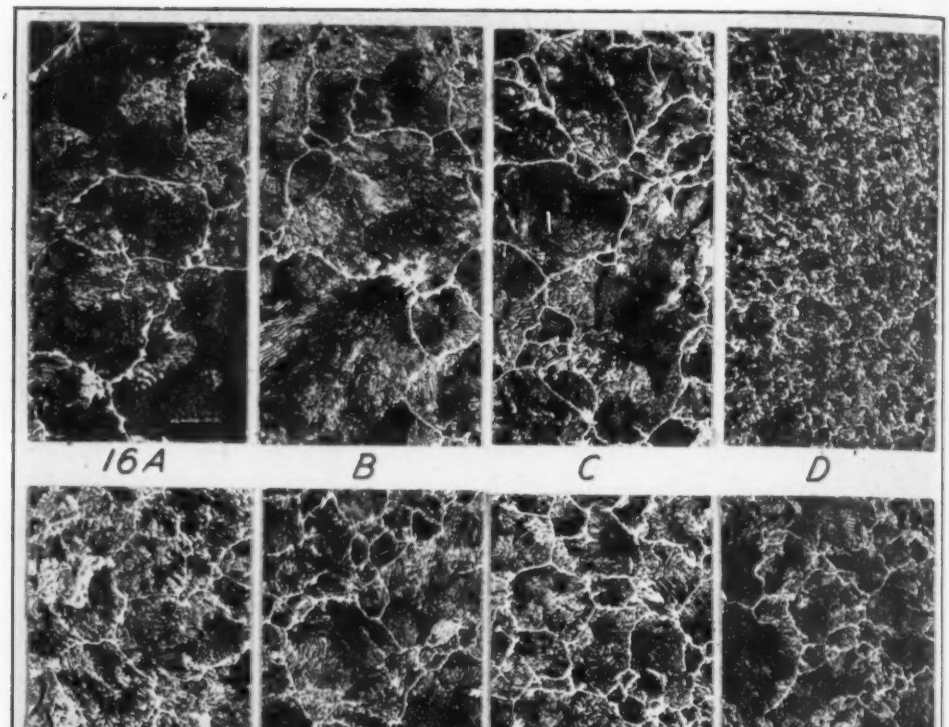


Fig. 16—Photomicrograph of Hypereutectoid Zone of Carburized Annealed Samples of Steels A, B, C, and D Cooled at a Rate of 220 Degrees Fahr. Per Hour to 1000 Degrees Fahr. × 100. Fig. 17—Photomicrograph of Hypereutectoid Zone of Carburized Quenched from 1500 Degrees Fahr. Samples of Steels A, B, C, and D Cooled at a Rate of 220 Degrees Fahr. Per Hour to 1000 Degrees Fahr. × 100. Fig. 18—Photomicrograph of Hypereutectoid Zone of Carburized Quenched from 1500 Degrees Fahr. Samples of Steels A, B, C, and D Ccoled at a Rate of 90 Degrees Fahr. Per Hour to 1000 Degrees Fahr. × 100.

and Rawdon,[1] of the Bureau of Standards, show that abnormal tool steels are more prone to have soft spots on water quenching. They state, however, confirming Merten[2, 3], that this tendency is overcome by the addition of brine or caustic to the quenching water. As we have throughout this investigation used a 4 per cent brine solution as a quenching medium, the question of proneness to soft spots is not a practical consideration, at least with us. A review of metallurgical literature on the normality or abnormality of steels after the McQuaid-Ehn carburizing test brings to light a difference of opinion as to what constitutes abnormality. McQuaid and Ehn[4, 5, 6] define abnormality as: (1), a finer and more irregular grain structure, and (2), a tendency toward breaking up the pearlite grains in the hypereutectoid zone to form massive cementite and free ferrite areas. Gat[7] found lack of correlation between variations in grain size and the tendency towards divorce of the pearlite. Herty, Larsen, Krivobok, Norton, Wiley, Sikes and Jacobs[8] state that they believe that there is no connection between variations in grain structure and tendency towards divorce of the pearlite. Sefing[9] states that he is able by high temperature quenching previous

[1]S. Epstein and H. S. Rawdon, "Steel for Case Hardening—Normal and Abnormal Steel"—Bureau of Standards Journal of Research R. D. No. 14, 1928, p. 423.

[2]W. J. Merten, "Fused Salt Baths to Prevent Soft Spots in Carburized Steel," TRANSACTIONS, American Society for Steel Treating, Vol. 7, 1925, p. 23.

[3]W. J. Merten, "Irregular Carburization of Iron and Iron Alloys, Cause and Prevention," TRANSACTIONS, American Society for Steel Treating. Vol. 9, 1926, p. 907.

[4]H. W. McQuaid and E. W. Ehn, "Effect of the Quality of Steel in Case Carburizing Results," *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 67, 1922, p. 341.

[5]E. W. Ehn, "Influence of Dissolved Oxides on the Carburizing and Hardening Qualities of Steel," *Journal*, Iron and Steel Institute, Vol. 105 No. 1, 1922, p. 157.

[6]E. W. Ehn, "Irregularities in Case Hardening Caused by Improperly Made Steel," TRANSACTIONS, American Society for Steel Terating, Vol. 2, 1922, p. 1177.

[7]J. D. Gat, "Normality of Steel," TRANSACTIONS, American Society for Steel Treating, Vol. 12, 1927, pp. 376-413. Discussion pp. 413-435.

[8]C. H. Herty, Jr., B. M. Larsen, V. N. Krivobok, R. B. Norton, R. E. Wiley, A. W. Sikes, and J. E. Jacobs, "Abnormality in Case Carburized Steels," Mining Metallurgical Investigations. Carnegie Institute of Technology U. S. Bureau Mines and Mining and Metallurgical Advisory Boards Cooperative Bulletin No. 45, 1929.

[9]F. G. Sefing, "Control of Normality in Plain Carbon Steels," Bulletin No. 13 Engineering Experiment Station Michigan State College of Agriculture and Applied Science, p. 927.

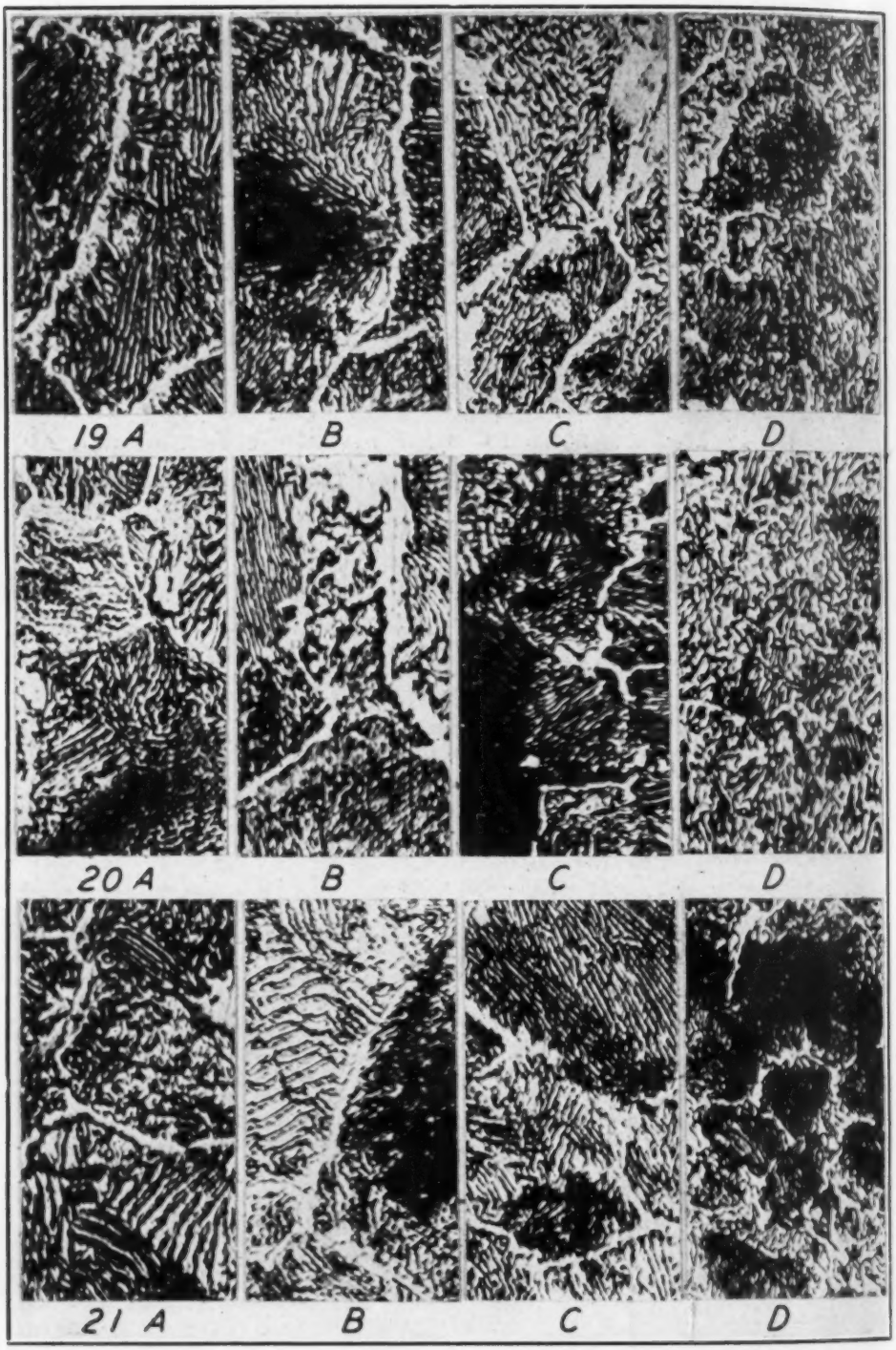


Fig. 19—Photomicrographs of Hypereutectoid Zone of Carburized Annealed Samples of Steels A, B, C, and D Cooled at the Rate of 220 Degrees Fahr. Per Hour to 1000 Degrees Fahr. × 500. Fig. 20—Photomicrographs of Hypereutectoid Zone of Carburized Quenched from 1500 Degrees Fahr. Samples of Steels A, B, C, and D Cooled at the Rate of 220 Degrees Fahr. Per Hour to 1000 Degrees Fahr. × 500. Fig. 21—Photomicrographs of Hypereutectoid Zone of Carburized Quenched from 1500 Degrees Fahr. Samples of Steels A, B, C, and D Cooled at the Rate of 90 Degrees Fahr. Per Hour to 1000 Degrees Fahr. × 500.

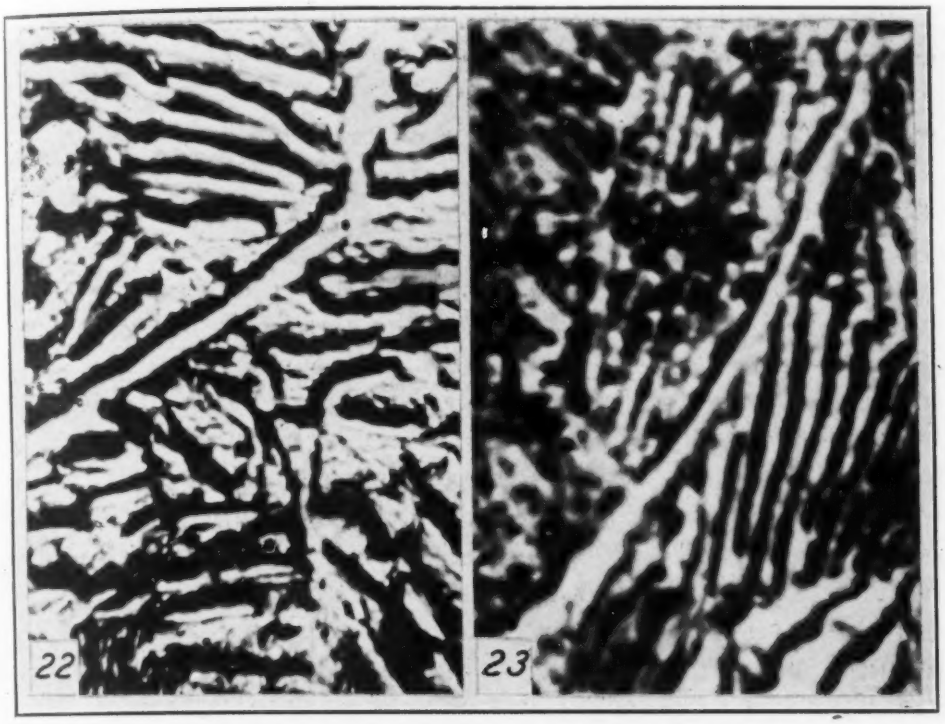


Fig. 22—Photomicrograph of Carburized and Slowly Cooled Tool Steel Illustrating Tendency Towards Divorced Pearlite at the Cementite Grain Boundary. × 2000.
Fig. 23—Photomicrograph of Carburized and Slowly Cooled Tool Steel Illustrating Regular Pearlite Formation at the Cementite Grain Boundary. × 2000.

to carburization to change an abnormal steel into a normal one. He does not clearly define whether he refers to the structural abnormality or grain size abnormality. Larsen and Sikes[10] check his results and conclude that structural abnormality cannot be changed by heat treatment previous to carburization but grain size abnormality may be modified. In making our test on the tool steels we have attempted also to obtain some information relating to the controversy.

The McQuaid-Ehn tests were conducted in the following manner: The samples were packed in a barium carbonate energized carburizing compound, heated to 1700 degrees Fahr, held for a period of 8 hours and cooled at the rate of 220 degrees Fahr. per hour to 1000 degrees Fahr. The samples were treated before carburization in the following manner:

The first series, no treatment. Annealed "as received" state.

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[10]B. M. Larsen and A. W. Sikes, TRANSACTIONS, American Society for Steel Treating, Vol. 14, 1928, p. 355.

The second series, quenched from 1500 degrees Fahr. in brine water.

The third series, quenched from 1600 degrees Fahr. in brine water. And in order to study the effect of the rate of cooling a fourth series, quenched from 1500 degrees Fahr. in brine and cooled at a rate of 90 degrees Fahr. per hour to 1000 degrees Fahr.

The grain size abnormality was considered apart from structural abnormality. Figs. 16, 17, and 18 are illustrative of the grain size of the first, second, and fourth series and Figs. 19, 20, and 21 are illustrative of the state of the pearlite of the first, second, and fourth series. The third series of obtained structures were not photographed as they were similar in structure to those of the second series. The deductions from the micro-examinations were that the grains of steels A, B, and C were of medium and regular size, that the grains of steel D were irregular in size ranging from smaller to larger than steels A, B, and C, that the grain size of steels A, B, and C. was not changed by the treatment given them prior to carburization or by the rate of cooling; that the grain size of steel D was changed by treatment prior to carburization but that the rate of cooling did not affect the grain size.

As far as the structural normality of the steels is concerned it would be difficult to say whether they are normal or abnormal. In certain areas the pearlite is well formed and in other areas the pearlite has a tendency to be divorced. These two states are illustrated in Figs. 22 and 23. Steel D had considerably more areas of divorced pearlite and could be considered as being less normal or more abnormal than steels A, B, and C. The structural normality of the steels was not modified by heat treatment prior to carburization or by the variation in the rate of cooling.

Part II

Design, Heat Treatment and Use of Dies Made from Steels A, B, C, and D

In the first part of the paper was discussed the physical characteristics of the steels used in the investigation and their behavior during a series of heat treatments. It is expected that these steels will exhibit the same properties and behave in the same manner, with certain modifications due to the increased mass of the steel, when they are formed into dies.

Die Design

The steels were machined into dies of the design shown in Fig. 24. Though primarily the die design is governed by the shape of the desired bolt there are certain modifications that are resorted to in order to improve the die life. For example, the forming of a small radius in the corners of the die had the effect of more than doubling the die life. A considerable increase in the die life was also obtained by rounding off the edge of that part of the die which forms the shoulder on the shank of the blank. When this edge was sharp it filed off the surface of the bolt wire and these chips lodged in the corners of the head of the blank causing rapid deterioration of the die. The principle of the design of dies with reference to heat treatment is well covered in a paper by Palmer.[11]

Heat Treatment of Dies

The dies were treated in a manner similar to the 1-inch rounds, by the hump method. Two quenching temperatures and two quenching times were used. A duplication of the treatment of the 1-inch rounds namely: First series, heated to 125 degrees Fahr. above hump, held 20 minutes and quenched in a 4 per cent brine solution. Second series, heated to 125 degrees Fahr. above hump, held 60 minutes and quenched in a 4 per cent brine solution. Third series, heated to 225 degrees Fahr. above hump, held 20 minutes and quenched in a 4 per cent brine solution. The temperature of the brine solution was held between 60-70 degrees Fahr.

By reason of the increased mass of a die over that of a 1-inch round the rate of heating to the quenching temperature must be considerably modified. It is of the greatest importance that the dies be heated in a uniform manner to the quenching temperature. It is generally necessary to obtain the proper rate of heating for each individual die shape. The correct rate of heating was obtained by the controlled temperature method as described by Hegel.[12]

A hole was drilled in the side of a die equidistant from the ends and 0.775 inch from the circumference and a thermocouple inserted in the hole. The hole was then carefully sealed. The

[11] F. R. Palmer, "Notes on the Relation of Design to Heat Treatment," TRANSACTIONS, American Society for Steel Treating, Vol. 14, 1928, p. 469.

[12] G. W. Hegel, "Heating of Steel by the Controlled Temperature Method," TRANSACTIONS, American Society for Steel Treating, Vol. 14, 1928, p. 377.

furnace control couple, by careful manipulation of the heating elements, was maintained within 15 degrees Fahr. of the couple in the die until the die passed through the critical range and on to the quenching temperature. The rate of heating as obtained by this means was used in the heating of the test dies. The test dies, in batches of four, were placed in an electrically heated furnace maintained at 1200 degrees Fahr., allowed to equalize and heated at the predetermined rate to the quenching temperatures.

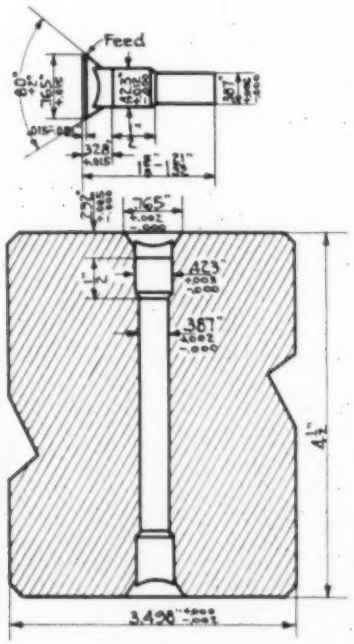


Fig. 24—Drawing Showing Dimensions of Die and Blank and Dimensional Tolerances.

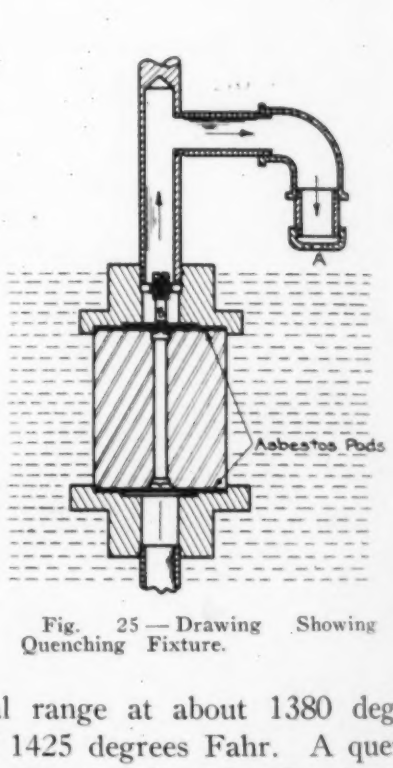


Fig. 25 — Drawing Showing Quenching Fixture.

The steels entered the critical range at about 1380 degrees Fahr. and passed through at about 1425 degrees Fahr. A quenching fixture slightly modified from that described by Cope[13] (Fig. 25) was used. A certain amount of experimental work had to be done in order to determine the diameter of the outlet A (Fig. 25) best suited to the quenching setup and the pressure of the 4 per cent brine solution entering the fixture. Regarding pressures, we had previously used water pressures as high as 80 pounds but with moderate success, the tendency being to produce a non-uni-

[13]L. S. Cope, "Hardening Cold Heading Dies," TRANSACTIONS, American Society for Steel Treating, Vol. 14, 1928, p. 51.

formity of hardness and case depth in the die. The end of the die at the water inlet had a considerably deeper case than the end of the die at the outlet end. This as well as varying hardness produced unequal life on the ends of the dies. The pressure was reduced to 15 pounds. This reduction of pressure and the addition of brine to water gave the highest and most uniform production from both ends of the dies. The dies were withdrawn from the quenching fixture while still warm. This precaution is usually taken by tool hardeners and we have always found it to be a commendable practice. Many failures of tools both after hardening and during service will result from cracks developed by cooling completely. Further in this regard we find it desirable to temper the dies as soon as possible after withdrawal from the quenching medium.

All the dies were tempered in an electrically heated furnace in which the air was circulated, at the temperature of 500 degrees Fahr. for a period of four hours. This tempering temperature was maintained throughout in order to keep this factor in the test constant. This treatment produced a uniform hardness of 57 to 60 Rockwell C on both ends of the dies.

It is of considerable importance to know within close limits what size changes take place during the heat treatment. The general tendency of a die of this design is for the hole to shrink which means that dimension 0.423 inch on Fig. 24 is decreased. As far as the steels A, B, C and D are concerned the decrease in this dimension amounted to less than 0.003 inch. The least movement was observed in steel C. When the extent of the movement is known allowances for these dimensional changes may be made in machining the dies. A certain amount of the decrease in the dimensions 0.423 and 0.387 inch on Fig. 24 were taken care of by a lapping operation. This lapping operation is in any case necessary in order to remove scale or surface decarburization. After this final sizing or lapping operation the dies are ready for use.

Bolt Wire

Considerable thought has been given to the production of dies, uniformly and correctly heat treated, from the steel A, B, C, and D. If, however, results as to die life are to be obtained the dies must be closely followed when in use. It has been our experience

Table VI
Effect of Surface Condition of Wire on Die Runs

Condition of Wire	Production* No. of Blanks	Heat Treatment	Steel
Heavy lacquer coat	3,600	Quenched from 225 degrees Fahr. above hump, held 20 minutes at heat	A
Light coat practically bright	18,500		
Clean Wire	12,700	Quenched from 125 degrees Fahr. above hump, held 20 minutes at heat	C
Oily Wire	3,300		
Clean Wire	26,000	Quenched from 225 degrees Fahr. above hump, held 20 minutes at heat	D
Oily Wire	1,300		

*Averages of ends of dies.

that as important as is the correct choice of the steel and the careful heat treatment of the dies, the most overwhelming factors are the wire and the machine operation. Unless these variables can be controlled within close limits the most carefully heat treated dies made from the highest quality steels will break down in a comparatively short time. These factors are the cause of the most misleading conclusions as to the best steel or heat treatment in a given case. The question of bolt wire is discussed; and some of the effects of defective wire are given in Table VI.

The size of the wire in relation to the size of the hole in the die is the first wire consideration. The nominal size for use in a die of this design (Fig. 24) should be 0.384 inch in diameter. The commercial cold drawn tolerance is 0.002 inch plus or minus. This tolerance means that the wire may vary 0.004 inch in all. The variation is too wide for the uniform die life to be maintained. The wire should preferably run with 0.001 inch plus and minus. An out of round condition of the wire is also to be avoided. Wire is occasionally received from the mills exceeding the maximum size of 0.386 inch. Unless this wire is eliminated by the machine operator, this oversized wire going in the die will score the die and necessitate it being removed from the machine for relapping.

The analysis of the wire has a considerable effect on the die life. Pure iron flows more easily and hardens less on cold working and consequently would be an ideal material with respect to prolonging the die life. An increasing carbon content, because of the effect of carbon on the flow and the hardness after cold work, materially decreases the die life. The wire used in these experimental dies was of the following analysis: Carbon 0.30-0.37, Manganese 0.50-

Table VII
The Effect of the Carbon Content of the Wire on the Die Life

Analysis of Wire	Production* No. of Blanks	Heat Treatment	Steel
C 0.36 Mn-0.61	21,000	Quenched from 225 degrees Fahr. above hump, held 20 minutes	A
C 0.11 Mn-0.40	37,400		

*Average of ends of dies.

0.80, Sulphur 0.050 (max.), Phosphorus 0.045 (max.). In order, however, to study the effect of the carbon content on the die life some dies were run using wire of the following analysis: Carbon 0.08-0.12, Manganese 0.30-0.60. A comparison is given in Table VII.

Manganese up to 1.25 per cent at least does not affect the die life when the carbon is under 0.20 per cent. Very successful die runs were obtained when using a wire of the analysis: Carbon 0.14-0.19, Manganese 0.70-1.00 per cent.

The physical properties of the wire in tension, especially the figure for the reduction of area cause a material lengthening or shortening of the die life. A high reduction of area value is very desirable because it guarantees a rapid flow of metal without rupture. A reduction of area value of no less than 40 per cent is preferred. The range of physical properties which gives the best results in the Carbon 0.30-0.37, Manganese 0.50-0.80 wire is ultimate stress in pounds per square inch 90,000-100,000, reduction of area 45-55 per cent. Unfortunately wire is shipped from the mills with reduction of area percentages as low as 20-25 even in wire which has been normalized or annealed before drawing. These low reductions of area values are often due to nonmetallic inclusions in the steel, overstrain in previous processing or overdrawing. Reduction of area values as low as 20-25 per cent, do greatly shorten the die life.

The surface hardness of the wire is of some importance; the hardness of the 0.30-0.37 per cent carbon wire ranges usually between 70-90 Rockwell "B". A variation in hardness within a short length of wire is very detrimental for if the wire is hard in one part of the circumference and soft in other parts of the same circumference the first upset (Fig. 1) will be formed of irregular shape. The most important factor of all is the surface condition of the wire. A rough seamy surface scores the die. The wire is inspected in this regard by lightly etching a short length in 1:1

hydrochloric acid, the etching reveals seams, slivers and other surface defects. Wire exhibiting these defects in a marked degree should not be used. A number of coatings have been developed by the wire manufacturers which are claimed to increase the die life by lessening the friction between the wire and the die. In many cases the use of these lacquer coats has improved the die life but in other cases the lacquer coat has the opposite effect. As a general rule a very light lacquer coat or a bright wire should be used when the die is designed to form bolts which have a sharp cornered head. Considerable trouble was experienced by us in using wires coated with a heavy lacquer especially where the lacquer becomes less viscous through the heat generated by friction between the die and the wire. The lacquer coat gathered in the corners of the die and it was impossible to fill out the corners and thus a defective bolt was formed. In order to use the die further it had to be taken out of the machine and the corners cleaned out.

Oils, both heavy and light, on the surface of the wire are very detrimental to die life when the die is of this design. The heat of friction is sufficient to cause minute explosions in the corners of the die when the first upset is forced into the corners of the die by the finishing punch. The difference of die life between that of clean dry wire and oily wire is shown in Table VI as is the effect of a heavy lacquer coat.

Machine Operation

The dies were used in a solid die double blow cold heading machine. Briefly the machine operates as follows: The wire is fed into the machine where it is sheared to the proper length (Fig.1). A finger picks up this wire and places it in front of the die where the cone punch gives the length of wire its first upset (Fig. 1). The cone punch recedes and at the same time raises on a transerve slide—its place being taken by the finishing punch. This punch advances on the ram and strikes the upset wire giving it a second upset and so forming the plow bolt blank (Fig. 1). The blanks are formed at a rate of from 55-65 per minute.

The operator holds in his hand the fate of the die. The length of the life of the die is dependent to a great extent on his skill. As the wire enters the machine it passes through two rolls to the shears. If these rolls are adjusted too tightly the wire is

apt to be scored. If the shears are blunt the wire will be pressed out of round. The cone punch gives the wire its first upset. The adjustment of this cone punch is important because if it strikes too hard it is difficult for the finishing punch to form the bolt and there is considerable excess wear on the die. Should the blow of the cone punch be too light the finishing punch has extra work to perform and it is probable that the corners of the die will not be properly filled out. As the finishing punch comes into action a mechanism called the relief attachment operates. Its function may be described as follows: When the wire length is first fed in front of the die the knock-out pin in the die, backs up the wire length. After the first upset in which the knock-out pin has backed up the wire length, the position of the knock-out pin is relieved so that it may recede a little when the first upset is struck by the finishing punch. This relieving helps the finishing punch to fill out the corners of the die. Too little relief may result in the corners of the bolt not being properly filled out. Too much relief will result in either the cracking of the die or the breaking out of the corners of the die. The thickness of the feed or flash on the type of blank we are dealing with (Fig. 24) has an important bearing on the die life for the feed acts as a cushion between the finishing punch and die. We normally operate with a feed of 0.025-0.035 inch which is on the maximum side of the tolerance of this dimension. This feed is controlled by the length of wire cut off for the blank. It is advisable for the operator to make his set up on an old die and after the setup is properly completed to substitute a new die. A good die may be ruined after a few blanks have been made if the machine setup is wrong.

The Results of Test Die Runs

When sufficient knowledge has been obtained so that the variables wire, and machine operation may be controlled within close limits then and only then can accurate information be obtained with regard to steel or heat treatment. Table VIII gives a summary of the test dies run under ideal conditions as to wire and machine operation.

Types of Failure

In the early stages of the investigation before we determined

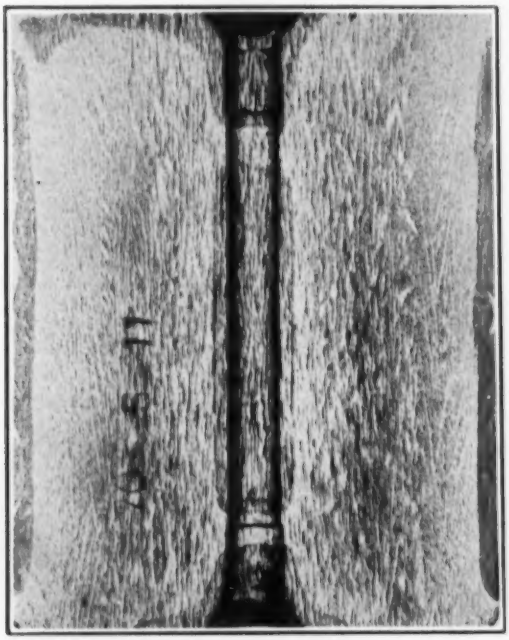


Fig. 26—Photograph of Die Cut Through the Center and Etched in Order to Show Break in Hardened Case of the Hole in the Die Which Forms the Blank.

Table VIII
The Effect of Steel and Heat Treatment on Production

Heat Treatment	Steel A	Steel B	Steel C	Steel D
Quenched from 125 degrees Fahr. above hump, held 20 minutes.	11,100	10,500	10,400	32,200
Quenched from 125 degrees Fahr. above hump, held 60 minutes.	.....	.....	20,100	40,800
Quenched from 225 degrees Fahr. above hump, held 20 minutes.	22,200	20,500	30,000	27,300

NOTE. The figures give the average blank production on ends of the dies.

the diameter of the quenching medium outlet A (Fig. 25) and the correct pressure through the quenching medium inlet, the dies failed early due to going oversize in the part of the hole in the die which forms the shank of the blank. This failure was due to lack of hardened case in the die at this point. (Fig. 26) When the quenching arrangements were uniformly satisfactory, the failures occurred by a breaking down in the corners of the die which forms the square of the head of the blank Fig. 27, or by chipping out of small flakes in the part of the die which forms the sloping part of the head of the blank. When we consider the design of the die we see that the breaking down of the corners of the square in the die would be the logical failure. This failure was a gradual process. It is

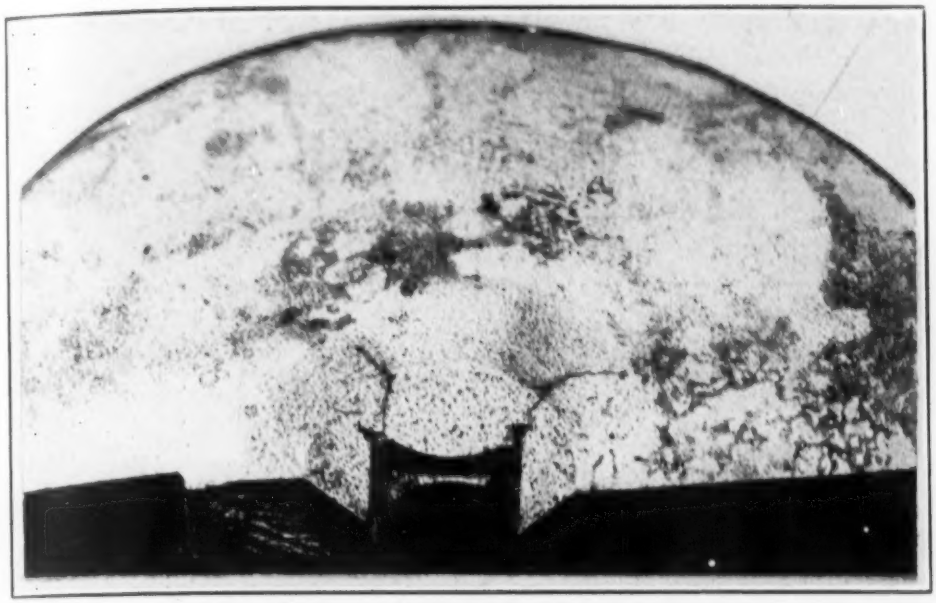


Fig. 27—Photograph of Split Die, Showing Breakdown of the Corners of the Part Which Forms the Square of the Blank Head.

exceedingly difficult, however, to account for flaking in the part of the die which forms the head slope on the blank at least prior to the break down of the corners. There seemed to be no correlation between the type of die failure and the steel or heat treatment.

Conclusion

To reiterate—the object of this investigation was to obtain the maximum production from a die used to cold form or cold head a type of bolt illustrated in Fig. 1. The maximum production was obtained from dies made from steel D quenched from 125 degrees Fahr. above the A_{3-2-1} point after holding for a period of 60 minutes at heat. It would be interesting to know why these results were obtained from this particular steel after the previously named heat treatment. In order to reach a conclusion, we will discuss the figures given in Table VIII. As far as heat treatment is concerned, quenching from a temperature in the neighborhood of 1625 degrees Fahr. doubled the life of dies made from steels A and B as compared with a quenching temperature around 1525 degrees Fahr. Steel C showed this increase to a more marked extent. This increase in production effected by increasing the quench-

ing temperature was noted by Wright.[14] Steels C and D also showed increased production by an increase in the holding time at the lower heat. The steels were not held at the higher heat for one hour because it was thought that the resultant decarburization and grain growth would nullify the benefits that might be obtained.

The production obtained from the dies made from steel D differed from the others with respect to the increased quenching temperature inasmuch as the production decreased when the tem-



Fig. 28—Curve Showing Relation Between Case Depth and Production.

perature was raised to the vicinity of 1625 degrees Fahr. What is the explanation of the different production values obtained from the steels themselves when given the same identical treatments? The steels were of the same high quality from the point of view of macrostructure and microstructure. It is maintained by some that a steel which shows a coarse grain when fractured after heat treatment is a preferred steel for cold heading die work, their explanation being that fatigue cracks proceed less rapidly through a coarse-grained structure. If this is so, why did not steel A give the best results? If a crucible melted steel has some remarkable inherent property, why did not steel B give at least better results than steel A? Did the higher production of steel D result from its abnormality? Wright shows slightly better results were obtained in cold heading ball dies made from an abnormal steel. We believe that none of these played a decisive part in determining the die life, but that the controlling factor was case depth. Fig. 28

[14] F. L. Wright, "High Temperature Quenching Treatment Applied to Cold Heading Ball Dies of Plain Carbon Tool Steel," TRANSACTIONS, American Society for Steel Treating, Vol. 13, 1928, p. 282.

is a graph in which the case depth of the 1-inch rounds is plotted against the die production. Whilst it must be admitted that this graph is not scientifically accurate, it does show that the general upward trend of the production did coincide with an increased case depth. In anticipation of an obviously justified question, we will answer that the dies were split in order to find a relation between the case depth of the 1-inch rounds and the case depth of the dies, and a very definite relation was found to exist between the two. The graph is, therefore, valid in this regard. An acceptance of the point brought out in the graph will readily explain why Wright found dies quenched from a comparatively high temperature gave better results, although he does not attribute his better results to this; rather he leaves the matter perfectly open.

Before concluding, we feel that we should attempt to explain, insomuch as there has been considerable discussion[15, 16] and material prepared on the subject of the variation of case depth found in different heats of steel apparently of the same analysis, why steel D hardened to a much greater depth than steels A, B, and C. We know the manufacturer of this steel made this steel with the object of increasing the case depth on quenching, but we are under the handicap of not knowing what he did to produce this quality. From observations made during the investigation, we believe that the inherent hardening properties in this heat at any rate are due to the increased manganese and chromium contents. The critical point determinations illustrated in Fig. 8 show that steel D has a lower Ar_{3-2-1} value and that the transformation was more sluggish. The steel when annealed under the same conditions as steels A, B, and C always showed a greater tendency to form sorbitic-pearlite (Fig. 4). Not only this, we were able by giving a steel a malleablizing treatment to malleablize steels A, B, and C without breaking down steel D.

To conclude, we will state that when we wish to obtain the maximum production from a given die, apart from bolt material and machine operation, the most important consideration is to obtain the right case depth. It is probable that the right case depth may be obtained mathematically, for it varies with the force of

[15]B. F. Shepherd, "Inherent Hardenability Characteristics of Tool Steel," TRANSACTIONS, American Society for Steel Treating, Vol. 17, 1930, p. 90.

[16]G. V. Luerssen, "Behavior of Carbon Tool Steel on Quenching," TRANSACTIONS, American Society for Steel Treating, Vol. 17, 1930, p. 161.

the impact and the distribution of the stresses on the die. Whether the right case depth is produced by varying the cooling rate, the quenching temperature, the holding time, or the chemical composition is a matter of choice.

DISCUSSION

Written Discussion: By James J. Curran, metallurgist, 2412 No. 2nd St., Harrisburg, Pa.

Mr. Jameson's interesting investigation has developed evidence as to several factors affecting die life, as follows:

1. Apparently insignificant variations in surface finish and cleanliness of the wire may exert tremendous effect on the life of the die.
2. In double end dies of the type used, extreme precautions are necessary to insure uniform hardening.
3. While high temperature hardening may lead to long life with one steel, this does not necessarily hold true for a similar steel from another maker, or possibly even from another heat by the same maker.
4. Our knowledge of the effect of normality on these tools is not yet complete enough to formulate a rule.

It is unfortunate that the steels used for this investigation did not show more pronounced extremes of normality, sufficient to furnish additional information on this angle of quality or "body" in tool steel. The author found it difficult to decide whether any of the steels were definitely normal or abnormal. Steel D, most interesting because of its superior life, is at the same time the most paradoxical. In Fig. 16D it exhibits a small grain size usually associated with abnormality, and the persistently fine grained fractures reported in Table IV indicate the same condition. Nevertheless, its higher manganese content indicates a tendency, at least, towards higher normality than the other three steels, and its greater depth of hardening at all temperatures is still further evidence of more pronounced normality. This relation between depth of hardening and normality is one indication on which all authorities appear agreed to date, viz., your present writer called attention to it for the first time in 1922,[1] and since that time it has been described by Epstein and Rawdon, and by Luerssen, in papers quoted by Mr. Jameson. For this reason, I would feel that steel D is more normal than the other three, and that its superior service tends to contradict the experience of others who have found abnormal steel better for cold heading dies.

Mr. Jameson's question as to why crucible steel B did not show at least better results than open-hearth steel A raises once more the question of "body", usually considered inherent in crucible steels, due to the generally higher grade, or at least more expensive, melting stock used. If normality has any relation to "body" in steel, then it evidently has nothing to do with the raw materials or melting stock used, as all degrees of normality may be

[1]Discussions following papers presented at the Tuesday morning technical session of the Annual Convention, Detroit, October, 1922. TRANSACTIONS, American Society for Steel Treating, Vol. 3, 1922, p. 317.

observed in tool steel of open-hearth origin, where little attention is paid to the grade of the melting stock. It is undoubtedly more dependent on the melting, refining, and pouring practices employed.

Written Discussion: By N. B. Hoffman, metallurgist, Colonial Steel Co., Monaca, Pa.

I quite agree with Mr. Jameson that the maximum production and life of cold heading dies are primarily dependent upon a number of principal factors; namely, die steel, die design, die heat treatment, bolt steel and machine operation. Cold heading dies could also be placed under two general classifications; namely, closed and open dies. I presume in this case, as per Mr. Jameson's manuscript, that he refers to closed dies only.

The chemical composition of the steels used as shown in Table I are primarily 1.00 per cent carbon tool steel. Sample "D", however, contains a small amount of chromium with a slightly higher manganese content and I assume contains no additional alloys even in similar small amounts. Mr. Jameson's statement relative to the value of carbon-vanadium steels producing satisfactory results are concurred in by a number of users of this type of steel, particularly for closed dies, and am sorry that he did not include a steel of this type in his investigation. The alloy in sample "D" evidently influenced the depth of hardness very much.

During the last several years I have performed many Ehn tests on 0.95 per cent carbon-vanadium crucible and electrically melted steels. Test samples of 1-inch round from each Ehn tested heat were finally quenched in cold water and acid etched to detect soft spots. Our results have shown that while the Ehn test was a fair measure of test relative to soft spots produced in the quench on electrically melted steel, such was not the case with the crucible melted product. In the latter product we have found many abnormal samples, as shown by the Ehn test, all of which hardened very satisfactorily and free from soft spots. My work in this case was concurred in by a large user of tool steel. The analyses of both steel types in this case were as near identical as possible. I cannot explain this differential, except to state that the crucible steel is an acid melted product, while the electric is a basic melted steel.

In properly hardening the dies much depends upon the type of quenching fixture. It is absolutely essential in anticipating maximum production from the dies that both die ends have a uniform hardness and of sufficient depth to prevent sinking. Many die failures, such as spalling and chipping, are primarily caused by sinking slightly due to insufficient depth hardness. To prevent spalling or checking of the surface core area of the dies, certain manufacturers have adopted the following procedure with very satisfactory results. The hardened dies are tempered at 410 degrees Fahr. in oil or other tempering media, after which both faces of the die are given a secondary temper to a light straw color by immersing in molten lead to a depth of $\frac{1}{8}$ inch or by setting on a hot plate. In any event the secondary temper should be performed as rapidly as possible, in that the depth of temper may be shallow.

As stated by Mr. Jameson, the size of the wire in relation to the size of the hole in the die is certainly one of the most important considerations. I have seen cases where they were using hot-rolled wire with a plus or minus tolerance of 0.005 inch. In this case the dies were bursted by forcing a wire

in the die which in some instances was as large or larger than the diameter of the hole in the die. A satisfactory wire tolerance should not be greater than 0.002-inch plus or minus and in no event should it be of greater diameter than the hole in the die. The gage tolerance of the wire can always be controlled, but the question of chemical analysis in many cases cannot be changed.

Written Discussion: By H. B. Pulsifer, metallurgist, Ferry Cap and Set Screw Co., Cleveland.

Mr. Jameson has given us an excellent outline of the problems involved in header die life. One is strongly impressed by the abundance of the variables, their intimate relation and the difficulty of counteracting that particular weakness that brings premature failure of a die. Mr. Jameson has followed through a sort of preliminary study on the primary variables. He should be warmly commended for frankly stating the results. We hope he will continue adjusting the variables until he can report a far greater die life.

Comments naturally drift toward suggesting more work; widening the range of the variables and attempting further combinations that logically

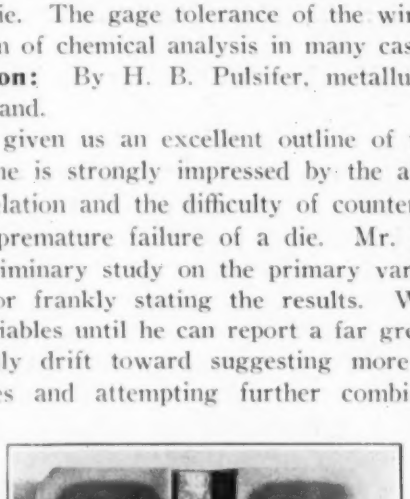


Fig. 1 — Etched Section Through 3.5-Inch Die.

promise greater die life. The problem is one of imposing magnitude and best treated by the statistical method. I have spent some time on the paper hunting for numerical quantities and average results. One finds "in batches of four" on the eighteenth page but no numbers are given in the tables to indicate if the results are averages or individual cases. Certainly, no unusual die life appears to have been attained.

Unquestionably, a fundamental truth has been stated when Mr. Jameson finds case depth of cardinal importance. The accompanying print shows an etched section through a 3.5-inch long die that failed after moderate service. The uniformity and depth of the case approximates the condition that we have found most satisfactory for our conditions. But, as Mr. Jameson well states, this is correlated with our particular variables of steel composition, treatment, wire and operations. The uniformity of the steel over long periods of supply is highly important. Given a uniform and suitable steel we find the

details of temperature and temper depend on die size, design, wire and operation exactly as Mr. Jameson enumerates.

After all, he has presented an excellent paper and we wish him many of those "considerable advances" as his experiments progress.

Written Discussion: By H. G. Keshian, metallurgist, Chase Companies, Inc., Waterbury, Conn.

As an analysis of the factors effecting the performance of cold heading dies, Mr. Jameson's paper is an addition of merit to our knowledge on the subject and worthy of serious consideration.

Referring to Figs. 2 and 3, as representing the macrostructures of the steels used for the tests, the author considers them of fair quality and entirely suitable for cold heading dies. While I do not wish to appear, in the least, as questioning this statement, I would be interested to know if Mr. Jameson has experimented with similar type of steels of more uniform structure or nearly free from porous centers than those indicated by these photographs. If so, what results did he get?

In Table VIII, the author has shown the effect of steel and heat treatment on production and seems to have used the same figures in the construction of the curve in Fig. 28, which shows the relation between production and case depth, but it is not stated or given in a little more detail as to how many dies each production figure represents. Although on the eighteenth page it is mentioned that the test dies were hardened in batches of four, I think it is desirable to know whether the production figures were the averages of four or more or less dies.

The reason of my stressing here the necessity of having in detail the production of each die in each group is this. From Figs. 2 and 3 it will be noticed that both of the etched disks show considerable porosity in the center. This condition, as it is well known, may increase or decrease in intensity or may entirely disappear at some distance from the etched end of the bar depending on which end of the ingot the etched disk represents. Therefore, if dies are made of these bars some of them may show more porous centers or segregation than others, and as this is the part of the die on which are concentrated the working stresses or the blows, this condition of varying degrees of porosity will introduce a chemical as well as structural variable which would obviously affect the die life, therefore, the test results. If this is so, it is, I think, important either to use bars of steel that are practically free from central segregation as revealed by etching the both ends or note in detail the production figures of each die in each group in a test of this nature where the general conclusion has to be based on the averages of each group of dies.

One of the other stimulating points of the paper is the place of alloy steels for this class of work. It has been the writer's experience for cold heading both ferrous and nonferrous materials, and I believe also the experience of others that plain carbon steels are to be preferred to alloy steels for dies. Wright,[2] for instance, says ". . . more than small traces of elements

[2]F. L. Wright, "High Temperature Quenching Treatment Applied to Cold Heading Ball Dies of Plain Carbon Tool Steel," TRANSACTIONS, American Society for Steel Treating, Vol. 13, 1928, p. 292.

such as chromium and tungsten will cause fatigue cracks to open up into flakings much sooner than steels free of those elements or steels containing only vanadium"; but Mr. Jameson's conclusion points to a different direction and he attributes the better performance of steel D to the presence of higher chromium and manganese than in steels A, B, and C. Therefore, we still have the question of alloy or carbon steels for heading dies before us.

In closing I wish to join the author in his recommendation for wire of proper physical characteristics for heading. It has been our experience that we were able to materially increase the life of header dies by insisting and getting softer wire, in many instances, than what seems to be practice of some steel mills to furnish. This is particularly true in the case of dies with sharp corners which cannot be filled with harder wire without excessive blows, thus subjecting the tool to greater stresses than is necessary.

Written Discussion: By G. Lofberg, vice-president, Uddeholm Company of America, Inc., New York City.

It is with some hesitation that I comply with the society's invitation to comment on Mr. Jameson's paper as I fully realize my inequality. Yet my views and observations might be of some benefit to students of the art rather than to students of the science of heat treating and this discussion is offered with the hope that it will aid in the continued progress of the cold forming industry.

Mr. Jameson's paper struck me immediately by its forceful introduction. The word's "relatively rigid body", in my estimation, embody the main principle for the successful heat treatment of cold heading dies.

Immediately after this revelation follows what could almost be considered the key to the entire cold heading operation and that is the five variables. Many steel men will remember when there was but one variable considered, die steel. Then came the recognition of variable No. 2—heat treatment, and this fact is largely responsible for the progress cold heading has made in recent years. A better introduction than this could hardly be found, or one more fundamental to the whole subject of cold heading methods.

I wish to congratulate Mr. Jameson for having thus clearly and simply brought out the two leading principles—"five variables" and "relatively rigid body". If they are constantly borne in mind by practical cold heading men they will mean a great deal for the progress of the industry.

After these introductory remarks, the first subject Mr. Jameson deals with is die steel. It is, undoubtedly, a generally recognized fact that for cold impact work the steel men are on the right road when they center their efforts along the line of straight carbon or near straight carbon steels. There are many reasons for this contention. One is the recognized impact fatigue resisting quality; another is the versatility; still another is the higher degree of uniformity from heat to heat. A good illustration of the versatility is Mr. Jameson's graph on page eight, illustrating the direct relation of heat and hardness penetration.

In regard to porosity tests, it might be worth mentioning that they are logical and commendable, although the good cold heading die steels on the market, no doubt, have all been tested in this respect by the manufacturers, yet

an extra check by the users would repay the expense of such tests many times over.

A very interesting subject and one much disputed is grain growth. The steel makers of today recognize this as one of their major problems and even have grain growth tests as standard equipment for every heat to be used for cold heading die steel. However, it is hardly the normal, reasonable grain growth which is so objectionable, but the question of uniform structure inasmuch as a good cold heading steel is apt to require at one time 1450 degrees Fahr. and at another time 1600 degrees Fahr. to yield the required depth of hardness penetration and it must give a good account of itself under all circumstances. For this reason it is desirable that the growth be the smallest possible with increasing heat. It is contended by many that the coarser grain is desirable for more resistance to the procedure of fatigue cracks, but even the most ardent admirer of this theory recognizes a saturation point beyond which increased growth results in disastrous brittleness.

On the subject of design, it is questionable whether the size of the die is always given proper attention, that is, the relation of die cavities to the wall within the scope that the available headers allow. This is a field of interesting possibilities not only for the die steel makers and the cold heading trade, but also for the cold header manufacturers.

Continuing with Mr. Jameson's heat treating, it is very commendable to find Mr. Cope's spray principle, which is an assurance of intense cooling effect. However, Mr. Jameson's idea about a reduced outflow of the quenching medium is interesting. It is not exactly a new idea but rather a new application. If I understand it correctly, this reduced outflow tends to act as a brake, forming, more or less, a solid column of water with the tendency to take the place of the mechanical spreader and, under any circumstances, aiding the spreader to force the fluid into every corner of the die cavity. It should be very interesting to compare the result of this arrangement with others like the counter spray where the water coming through the die is met by another stream of lower and properly balanced pressure.

Personally, I have advocated high pressure to intensify the cooling effect and drive the case to the desired depth at lowest possible temperature. On the other hand, the low pressure has given superior results in several well equipped shops in recent years and is now maintained by many as being superior to the high pressure.

The next subject of particular interest in this discussion is the die failures. From Picture 27 one would judge the "natural death" failure to be one of fatigue enhanced by sinking, the sinking in other words being the primary reason. This condition, however, can hardly be avoided, for if the case is driven deeper to obviate the sinking, the die will fail still earlier as is apparent from the eighth page and explained in Fig. 9.

As for the chippings in the sloping part, they might be due to localized stresses from without, but more likely from within. It is not impossible that they are caused in the hardening by steam pockets or air bubbles not removed fast enough by 15 pounds pressure.

In Mr. Jameson's conclusions, there are several questions which it might be of interest to reply to with the aid of a cold heading code which I have

formed as a result of years of observations and contact with the cold heading trade, thus getting an outside comparison with Mr. Jameson's conclusions. This code consists of six rules:

1. Cold heading die life is primarily a question of resilience.

   (A) A case that is deep enough to prevent sinking, yet shallow enough to allow resilience (give.)

   (B) A core that is equally well-balanced for perfect shock absorbing qualities. (Both size and structural considerations.)

   (C) A transition stage that assures a smooth blend of the case in the core.

2. The case is paramount for good results and varies for each individual job according to its size and design.

3. Once the correct case is found, die life can be increased to a certain degree by other conditions of which the most important is the draw; both time and temperature to be considered.

4. Obtaining the correct case is a matter of heat conduction and, consequently, depends on the three factors:

   (A) *The conductor itself*, which actually embodies two factors—analysis and the physical character of the die steel, both of which for the sake of simplicity might be termed—*Density*.

   (The effect of analysis on the $Ar_{3\text{-}2\text{-}1}$ point, or the atomic fluidity range is clearly shown by Mr. Jameson in his graph No. 8 on the seventh page. Physical character has reference to other factors like deoxidation, nitrogen and slag inclusions, etc., affecting the physical homogeneity, thus the conductive qualities of the steel.)

   (B) *The volume of heat applied*—that is the sum total of temperature and time.

   (C) *The intensity of cooling effect*—that is the sum total of temperature, pressure and density of the quenching medium (the volume of cooling effect).

5. Intensified cooling is the best means of increasing penetration and high heats should be resorted to only as a second line of defense for particularly deep cases.

6. The correct case is best developed by studying die failures and modifying subsequent treatments as the failures would indicate. (Sinking as a rule indicates too shallow case. Flaking and breaking out may indicate too deep case. Wear is mostly a question of drawing. These rules, however, are only approximate.)

With these points in mind it might be of interest to discuss Mr. Jameson's conclusions.

He first offers a "why" as explanation to "D's" success. The reply by this code, points 2 and 4, would be:

The reason why steel "D," quenched at 125 above Hump, after being held there for 60 minutes, gave the best results is due to the fact that the ideal case, or rather nearest ideal case, was obtained as a result of that

particular steel density, plus that particular heat, plus that particular cooling effect (45 degrees and 4 per cent brine and 15 pounds pressure.)

The next matter for discussion is the behavior of "C" and "D"—the former decreased—the latter increased die life as the quenching temperature was raised to 225 degrees above the Hump. This agrees very well with points 2 and 4 above. In other words, "C" was receding from the ideal case, getting constantly deeper into rigid zone, whereas "D" was in the sinking zone and approaching the ideal case.

The reason, according to point 4, was that "D" with its higher manganese and chromium contents was denser and, therefore, reached the ideal case depth at a lower temperature.

That my conclusions, independently reached, coincide with Mr. Jameson's opinion, to my mind eliminates the only criticism that could be made on his excellent paper, i. e., that his experiments were confined to individual dies rather than to averages of a great number.

In conclusion I wish once more to thank Mr. Jameson for this paper, which I feel has earned for itself a place as a milestone in the progress of the cold forming industry. Its greatest benefits are probably to be found in the fixing of responsibility between the steel maker and the steel user, and the stimulating of future efforts towards ever increasing efficiency by all those having the future of the cold heading industry at heart.

Written Discussion: By W. I. McInerney, superintendent, heat treating and cold drawing departments, Pittsburgh Crucible Steel Co., Midland, Pa.

The author has contributed an excellent paper on the very interesting subject of cold heading die life. He has shown, in general agreement with other writers on the same subject that: The reasonable expectancy of die life is greatly increased by a time-hi-temperature application, followed by pressure cooling using a refrigerated solution.

The analyses of the four steels A-B-C-D are not sufficiently dissimilar to select any particular one above the other, and the method of manufacture did not prove important, all were passed as being of "fair quality entirely suitable for use" after macro and micro tests, further the McQuaid-Ehn tests failed to show any particular superiority.

Table II introduces another variable that perhaps could be dispensed with, by having all the steels in the same approximate structural condition after annealing.

The various elements that increase depth hardness have been used with widely varying results. Where, theoretically, increased depth was desirable and obtained, the accompanying characteristics were not all satisfactory. The final solution to many individual problems has been to secure the correct combination of analysis and treatment.

The hazards tending to shorter the life of the ideal die, are so many compared to the precise methods used in the selection of the steel for, and the treatment applied to duplicate the ideal die, that it is quite evident that these destructive factors should be more thoroughly eliminated or brought under control to diminish the number of causes of failure.

A careful study of the paper only emphasizes the foregoing, and any attempt to answer some of the questions asked by the author in his conclu-

sion only reverts to the possibility of some of the destructive factors cited, any of which could have been a contributing cause of failure.

R. E. CHRISTIN:[3] Mr. Jameson is to be congratulated on his paper. He has aroused interest in the industry. Everybody interested in bolt manufacture is interested in the problem he has brought up in this paper.

I would like to ask Mr. Jameson in what manner that square was impressed in that die. I believe that a solid die with a square is quite a problem and the failure of that die is dependent in some way on the manner of sinking that impression. At least, that is our experience. We have found that broaching is really out of the question. There are other ways of doing it. I would like to have Mr. Jameson's view on the subject.

H. J. FRENCH:[4] I think it might be interesting, provided Mr. Jameson has time, if he enlarges upon the reasons for the superiority of carbon steels as against the alloy steels as classed for cold heading.

A. S. JAMESON: Mr. French asks us to enlarge upon the reasons for the superiority of carbon steels as against the alloy steels as classed for cold heading. Alloy steels, that is, steels with an appreciable content of alloy or alloys which increase the depth of hardening, except perhaps in a very few instances produce too deep a case in the die. With a very deep case or when hardened completely through the die fails early due to excessive rigidity. The softer core of the die seems to act as a cushion and absorbs the impact shock. Fig. 28 of the paper illustrates the effect of the case depth on the die life.

Author's Closure

We acknowledge our indebtedness to those who have contributed to the discussion of the paper and are gratified to find that in the main they are in agreement with our views on the subject of cold heading die life.

We wish to thank Mr. Lofberg for his kind comments and for the statement of his cold heading code.

As to the point raised by Mr. Hoffman, in regard to wire size, we believe it to be very true. In our preliminary work we found some wire 0.003-0.004-inch out of round and on one occasion we attempted to use wire which was 0.006-inch oversize. In this particular case the die was not burst but the corners of the square of the die were so severely cracked as to render the die useless in a comparatively short time.

Mr. Pulsifer as well as Mr. Keshian makes an inquiry as to the number of dies used in obtaining the production figures. In one case the figure represents an average of four dies while in all other cases the averaged figure was obtained from eight dies. In all, about one hundred and fifty double ended dies were used. As each end was considered as a separate entity this would mean that three hundred values were available for averaging.

Mr. Keshian raises an interesting point in mentioning the effect of the macrostructure on the die life, he also ties this in with the production figures. In answer to Mr. Keshian's question as to the effect of porous centers

[3]Metallurgist, Columbus Bolt Works, Columbus, Ohio.

[4]Metallurgist, Research Laboratories of The International Nickel Co., Bayonne, N. J.

on the die life, we find that within certain ranges the die life is not effected materially by the chemical segregation. We think it is a problem and will be a problem for some time in the cold heading industry to say definitely whether this or that porosity is detrimental in this or that application. We believe from our experience that the amount of porosity found in the bars we used is not detrimental to the die life. It should not be construed, however, that we would sanction the use of porous steel; in fact we will illustrate (see Fig. 1) a case where the porosity of the steel was an important factor.

We do not believe that our production figures were affected by the macrostructure of the bars we used. We chose our bars carefully, taking macro disks from both ends of the bars, knowing, as Mr. Keshian implies, that one end of a bar may represent the upper half of the ingot and, therefore, tend to show more porosity, due to chemical segregation, than the other end which would represent a lower cross-section of the ingot. Bars which showed end to end variation were not used in the test.

Mr. McInerney and Mr. Keshian refer to the alloy contents of steels used for cold heading work. We are not aware of any conclusive work on the effect of small quantities of alloys on the life of cold heading dies. It has been stated by one or two writers that traces of alloying elements are detrimental to die life, these statements are not, however, supported by evidence and are not sufficiently qualified. In expansion of this, suppose we admit the correctness of the findings of our paper and assume that for a particular application we must have a deep case. We show that the case depth of the steel we are using can be increased by increasing the quenching temperature or the time of holding at heat before quenching. Is this procedure, however, as desirable a way of obtaining a deep case as using a steel with a low alloy content? On the other hand in making up dies for heading very small articles the case must be as shallow as possible, in this instance a steel containing small alloy contents would be undesirable.

Mr. Curran deals at length in his discussion with the subject of normality and abnormality and its direct and indirect (i. e., effect on case depth) effect on the die life. We have not conceded that normality or abnormality had any measurable direct or indirect effect on the life of the dies described in our paper. We are not prepared to accept the unproved statements of writers that abnormal steels are better for cold heading dies. It may be that normality or abnormality have some indirect bearing on the die life. We are ourselves very interested in this subject and wish at some time to investigate this with reference to cold heading. We realize, however, the difficulty of the problem which becomes even more complex when we consider that normality or abnormality are not in themselves states but merely evidences of certain states or conditions in steel.

Answering Mr. Christin's question, the square was formed by broaching. We agree with Mr. Christin that broaching is not, perhaps, altogether desirable. We know that the metal is severely strained in broaching even to the extent of actual fractures in some instances. In this regard when making a die where broaching of this nature has to be done we find it desirable to see that the tool steel has a correctly annealed structure that is the structure consists largely of ferrite and spheroidized cementite. Other means of

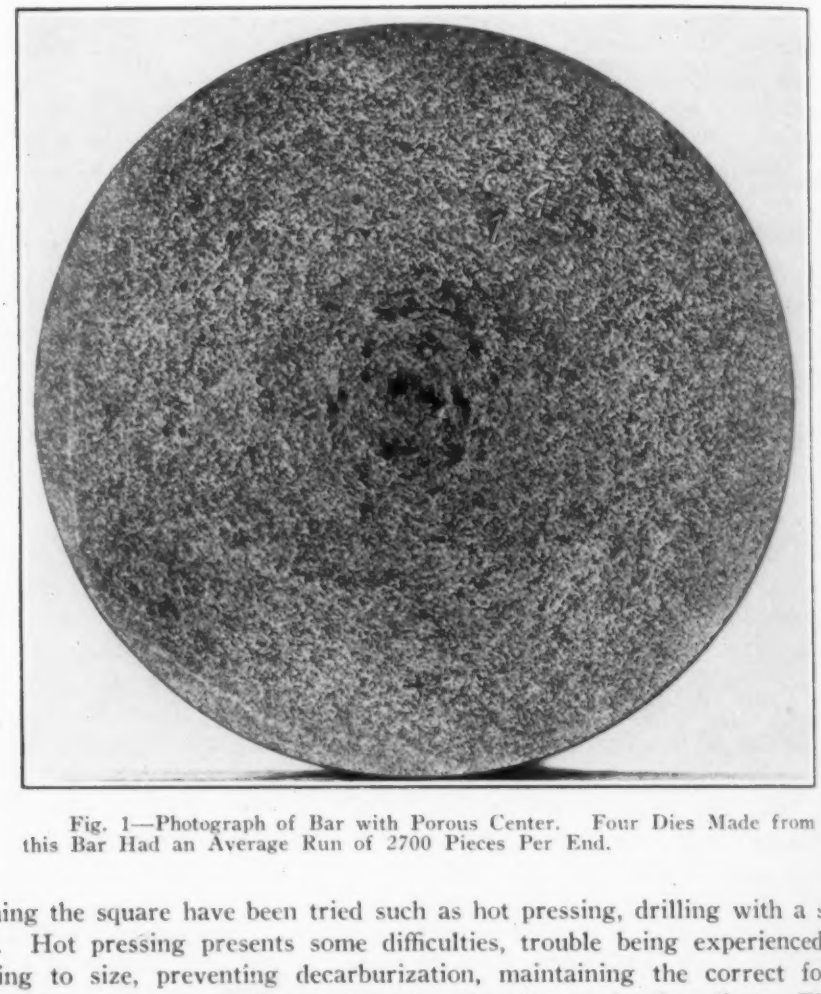


Fig. 1—Photograph of Bar with Porous Center. Four Dies Made from this Bar Had an Average Run of 2700 Pieces Per End.

forming the square have been tried such as hot pressing, drilling with a square drill. Hot pressing presents some difficulties, trouble being experienced with holding to size, preventing decarburization, maintaining the correct forming temperature so as to obtain the desirable direction of the flow lines. Though, we do not have any values available we did not obtain results which would compensate for the extra work on dies which were normalized in order to relieve the strains before hardening, however, we do not consider our work extensive enough to draw very definite conclusions.

VARIOUS HEAT TREATMENT FURNACES USED IN THE MANUFACTURE OF TELEPHONE EQUIPMENT

By W. A. Timm

Abstract

The manufacture of telephone equipment is a complex industry as more than twenty-one thousand different kinds of apparatus, constructed from approximately one hundred and twenty-seven thousand piece parts and using practically all of the known metals and non-metals, are produced. Many of these parts require heat treatments.

The purpose of this paper is to describe the various types of furnaces which are used by the Western Electric Company, Incorporated. Consideration is given to the procedure used in selecting heat treating equipment for proposed processes. A number of typical furnace installations, which illustrate the wide range of furnace equipment in telephone manufacture, are described in detail.

Classification of Uses for Furnace Equipment

THIS furnace equipment used in the manufacture of telephone equipment can be divided into three general classes. These are the furnaces used (1) for manufacturing processes, (2) tool room equipment, and (3) laboratory furnaces. Of these groups, the first mentioned is many times the largest. On account of manufacturing a large number of piece parts of a very diversified nature, the requirements placed on the various furnaces are quite different, and the types of furnaces provided are likewise very different in size and design.

A large number of furnaces are used for the manufacture or fabrication of metals. A number of furnaces are in service for the melting of lead, secondary copper, magnetic alloys, and precious metals. Others are used for heating billets of various kinds, previous to rolling and for annealing operations required during subsequent cold rolling operations.

During the process of making magnetic piece parts from sheet stock, many of the parts require hot forming after the blanking opera-

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, a member of the society, is development engineer, Hawthorne Works, Western Electric Co., Chicago. Manuscript received June 11, 1930.

tion. Special furnaces are being provided for such cases, to insure that the magnetic quality of the parts are not impaired by overheating.

Many piece parts require heat treatment after they have been completely formed and machined. Chief among these are the magnetic parts, which require very careful packing and treatment to insure that the desired physical properties will be obtained. Other parts are annealed for purely mechanical reasons, in order to facilitate subsequent drawing, or forming operations.

The furnaces used in the tool room are of standard types, which can readily be purchased on order from various furnace manufacturers. The reason for this is that during recent years the heat treatment of tools has been thoroughly studied by furnace suppliers, and furnaces for applications previously considered quite special have now been standardized.

Likewise, the laboratory furnaces which are used for routine tests are standard types.

Procedure Used in Selecting Furnace Equipment

It has not been found possible to set up a definite procedure to cover the development or purchase of special furnace equipment. On account of the varied uses for which the furnaces are required, all proposed furnace applications are given separate consideration.

A careful analysis is first made of the characteristics desired for the proposed materials or the piece parts under consideration. It is of the utmost importance that all specification requirements be met, as the success or failure of the particular furnace installation depends upon this factor as much as the economic factors as no degradation in quality is acceptable. Every proposed furnace installation is also carefully studied to determine if it is a safe piece of equipment for workmen to operate and if working conditions surrounding the furnace will be healthful.

After these conditions have been satisfied, attention is given to cost considerations. It is essential that the cost of operation of a particular furnace be low, and compete with similar type of equipment already in operation and with the standard types offered by furnace suppliers. Such economic studies always include first cost of the equipment, and a consideration of estimated operating and maintenance charges.

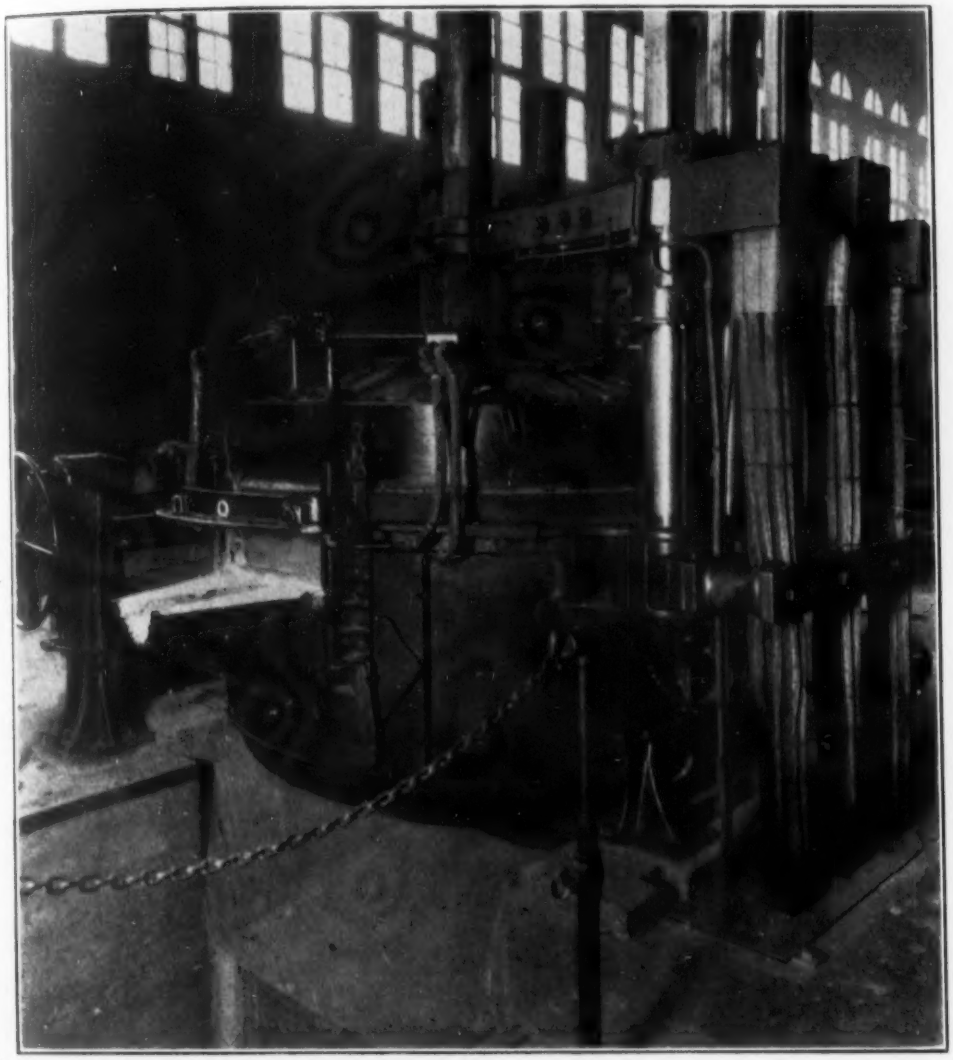


Fig.1—Electric Arc Furnace, Pouring Side.

The permanency of the use of the parts requiring heat treating equipment is next considered. The type of equipment selected depends, in a large measure, upon whether the equipment will be required over a period of years, or whether it would be needed for only a relatively short period of time.

The kind of energy to be used in heating a furnace is not given undue importance, since it is the purpose of the engineers to provide the type of fuel best suited for the installation under consideration. Accordingly, no effort has been made to standardize any heating medium such as oil, gas, or electricity. On the contrary, in some

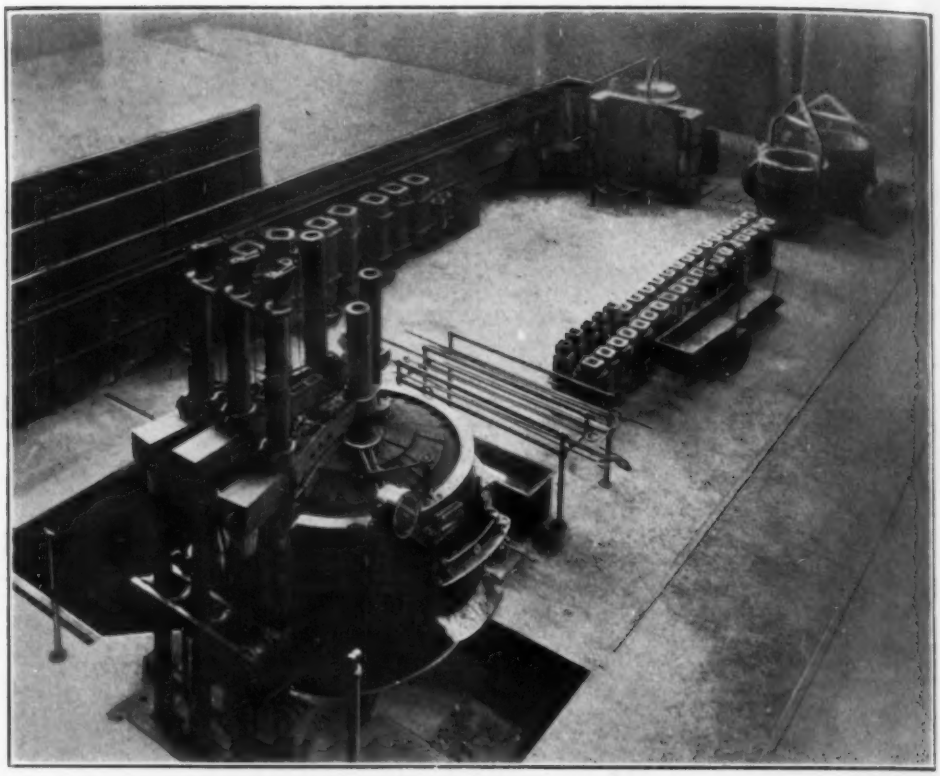


Fig. 2—Electric Arc Furnace, General View.

of the heat treating buildings, numbers of oil, gas, and electric furnaces are located practically side by side.

One factor, however, having bearing on the type of fuel selected, is the possibility of energy savings through the use of recuperative principles incorporated in the design of the proposed furnace or by the use of a separate recuperator used in conjunction with the furnace.

Consideration is also given to the appearance of any proposed furnace installation and its effect upon the general appearance of the room in which it is to be placed. Considerable attention is given to preliminary equipment layouts, and to modifications of design which will improve the general appearance of the proposed structure.

Types of Furnaces Used by the Western Electric Co., Inc.

The types of furnace equipment used by this company are many, since they were selected on the principle that the furnace provided must be the one best suited to the work. Accordingly, these furnaces

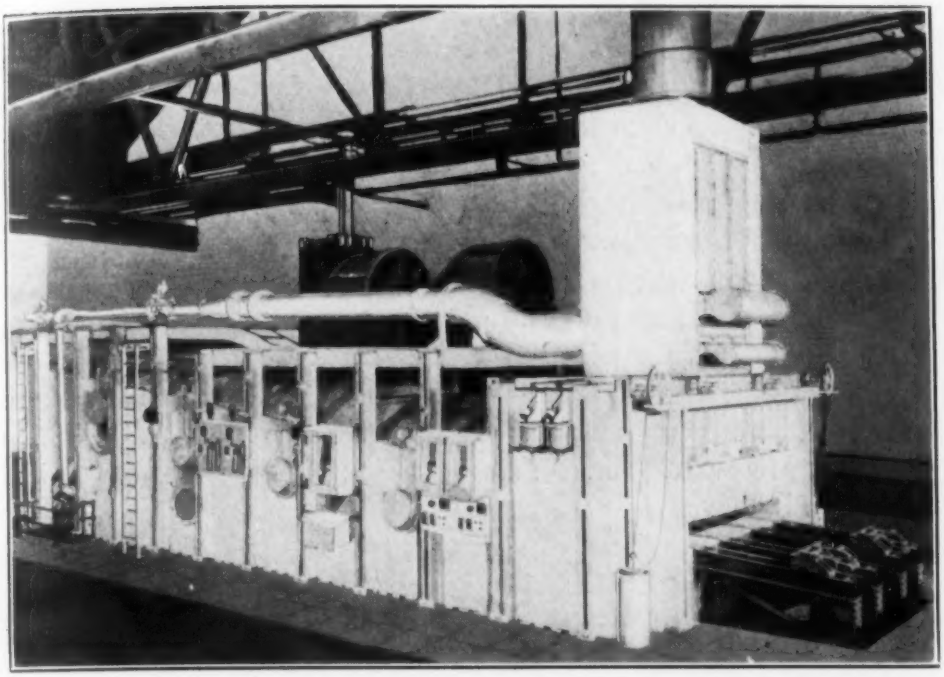


Fig. 3—Furnace for Heating Copper Billets, Prefatory to Rolling into Copper Rods.

are the product of different furnace suppliers. They are special, at least in some measure, as the supplier's experience in design and construction has, in most cases, been supplemented by the knowledge of the company's engineers in regard to the specific requirements to be placed on this equipment.

Many batch type furnaces are used. Most of these are single-door furnaces, although some are of the double-door type. The smaller batch type furnaces, in which small batches are heat treated, are loaded manually, usually with the aid of a monorail and hoist to the furnace door. Other larger types are loaded with battery trucks provided with elevating arms by which a number of boxes can be charged into the furnace at one time. Batch type furnaces are used only where the nature of the materials or parts handled make it imperative that they be handled as separate units. These furnaces are likewise used where a separate furnace is required to meet the demands of a part, having a continued, though relatively small, production.

For the hot forming of magnetic parts, rotary hearth furnaces have been found most satisfactory. This type of furnace is well adapted for the magazine feeding of small parts on to the hearth,

where they reach the required temperature in one revolution of the hearth, and are then in a position for the hot forming operation. These furnaces are connected with the punch press by electrical or mechanical links so that the stroke of the forming press automatically feeds the parts into the furnace. Magnet hardening furnaces are likewise made on this same general design.

Car type furnaces are also used. Several 160 K.W. to 200 K.W. car type recuperative furnaces have been installed for the heat treatment of materials and piece parts used in magnetic circuits. These furnaces are particularly well adapted for operation where a number of different kinds of charges, each requiring a different heat treatment, follow each other. They have been very successful in their cost of operation and in meeting the close temperature required for this product.

Continuous furnaces of the pusher type, and also continuous belt types, are also used for widely different purposes. These furnaces are satisfactory where the materials to be heat treated over a period of time are practically identical, so as to enable the scheme of continuous operation to be used.

Description of Typical Furnaces

In order to illustrate the usage of furnace equipment by this company, five furnaces will be briefly described. These furnaces are typical of other furnace installations.

Electric Melting Furnace

The melting of ferrous materials is performed in the electric arc furnace shown in Fig. 1. The materials made in this furnace are cobalt magnet steels and the various grades of ductile nickel-iron, chromium-nickel-iron alloy and brittle nickel-iron alloy, which are known as permalloy.

This furnace is circular in form with removable roof supported in a circular water-cooled channel section. It is basically lined with magnesite brick for the side wall and a "burned in" magnesite bottom. The roof is of silica brick as well as the few upper courses of brick in the side wall. The power is supplied to the charge by three 8-inch graphite electrodes. This installation is somewhat different from the usual one in that the power is supplied to the electrode arms by cables parallel to the electrode masts which are suspended in an un-

derground tunnel from the substation. A general view of this furnace and the arrangement of the casting floor is shown in Fig. 2.

The power is supplied to the substation at 13,200 volts. The usual transformers, reactors, switches, and other auxiliary equipment is used. The transformer is rated as 1200 K.V.A., 12 tap, 13,200 volt, 3-phase, 60 cycles. Power is supplied to the furnace electrodes at potentials ranging from 90 to 165 volts.

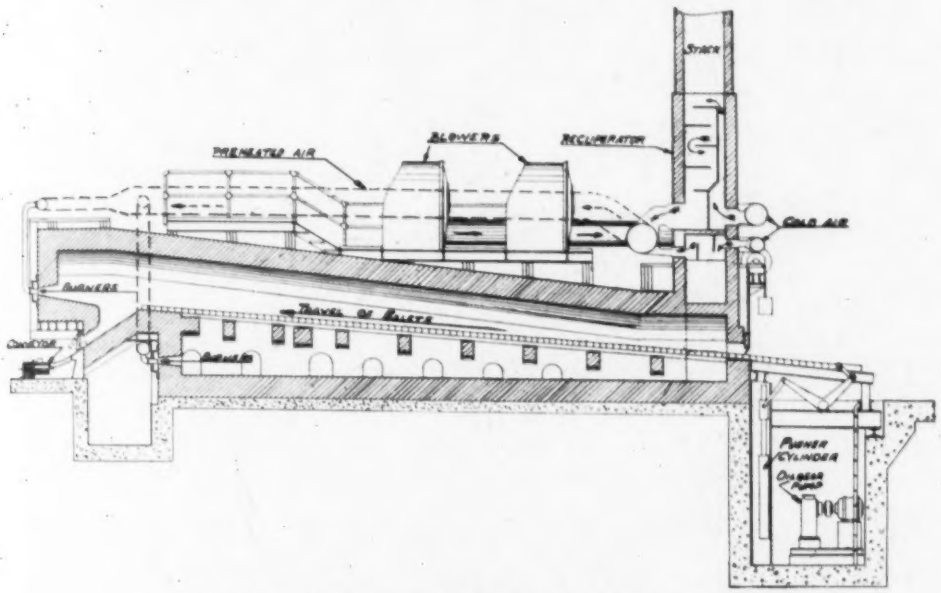


Fig. 4—Schematic Diagram, Showing Principle of Operation.

Normally five heats of 6000 pounds each are made each shift with a K.W.H. consumption between 470 and 520 K.W.H. per net ton depending, of course, on the previous temperature of the furnace, the type of charge, the material to be produced and other variables.

Furnace for Heating Copper Billets

The furnace shown in Fig. 3 was recently installed for heating copper billets preparatory to rolling into copper rods, which are subsequently drawn into wire. This is the largest furnace now installed in the Western Electric Company. It is approximately fifty-two feet long, and seventeen and one-half feet wide. The height of the furnace proper at the charging end is eight feet, while the top of the recuperator, located at this end, extends to twenty-one feet above the floor. The height of the discharge end is about eleven feet.

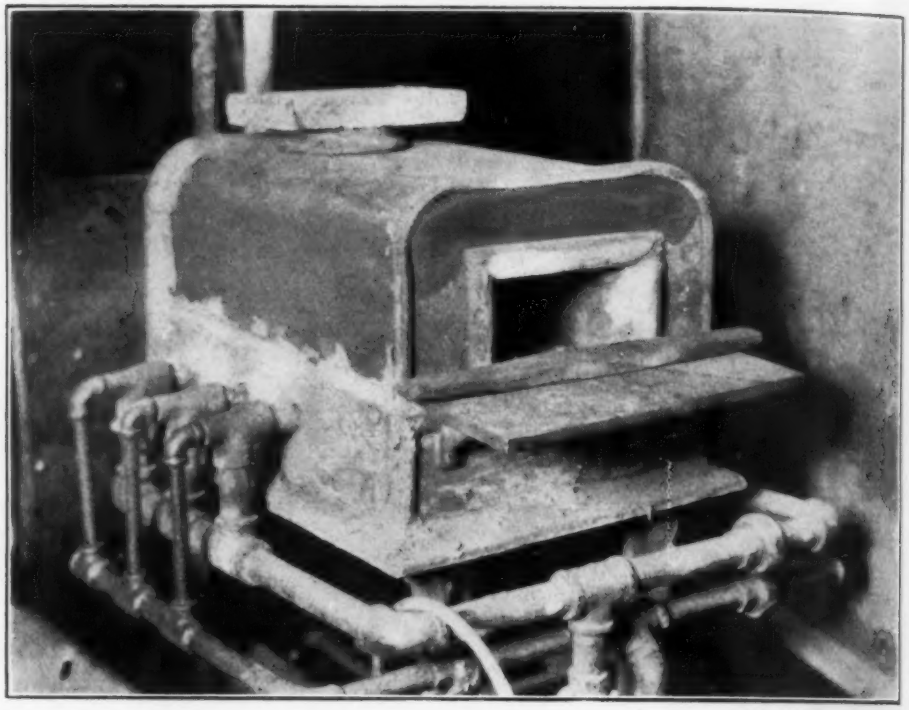


Fig. 5—Gas Fired Furnace Formerly Used for Heating Receiver Cores Previous to Hot Forming.

Two sizes of copper billets are heated in this furnace. These billets are either 54 or 60 inches long, by 4 inches wide, and 4 inches thick, and weigh 225 pounds and 250 pounds, respectively. Two rows of billets are fed alternately into the furnace by the charging device shown in the foreground. These billets are pushed sideways up inclined rails, where they are heated by the hot blasts from the two sets of oil burners, which are directed upon their top and the bottom surfaces. In this manner they continue to move up the inclined rails through the furnaces, always coming toward the two sets of five burners, and steadily increasing in temperature. They attain the predetermined temperature when they have reached a point several feet from the burners. They then slide down an incline to a conveyor located just outside of the discharge end of the furnace, which carries them endwise to the first pass of the rolls.

The location of the blowers and recuperator on the top of the furnace can be seen in Fig. 3. A schematic diagram of this furnace, illustrating the principle of operation is shown in Fig. 4.

This furnace burns fuel oil at the rate of about 200 gallons per hour, or approximately 5½ gallons per ton of billets heated. This

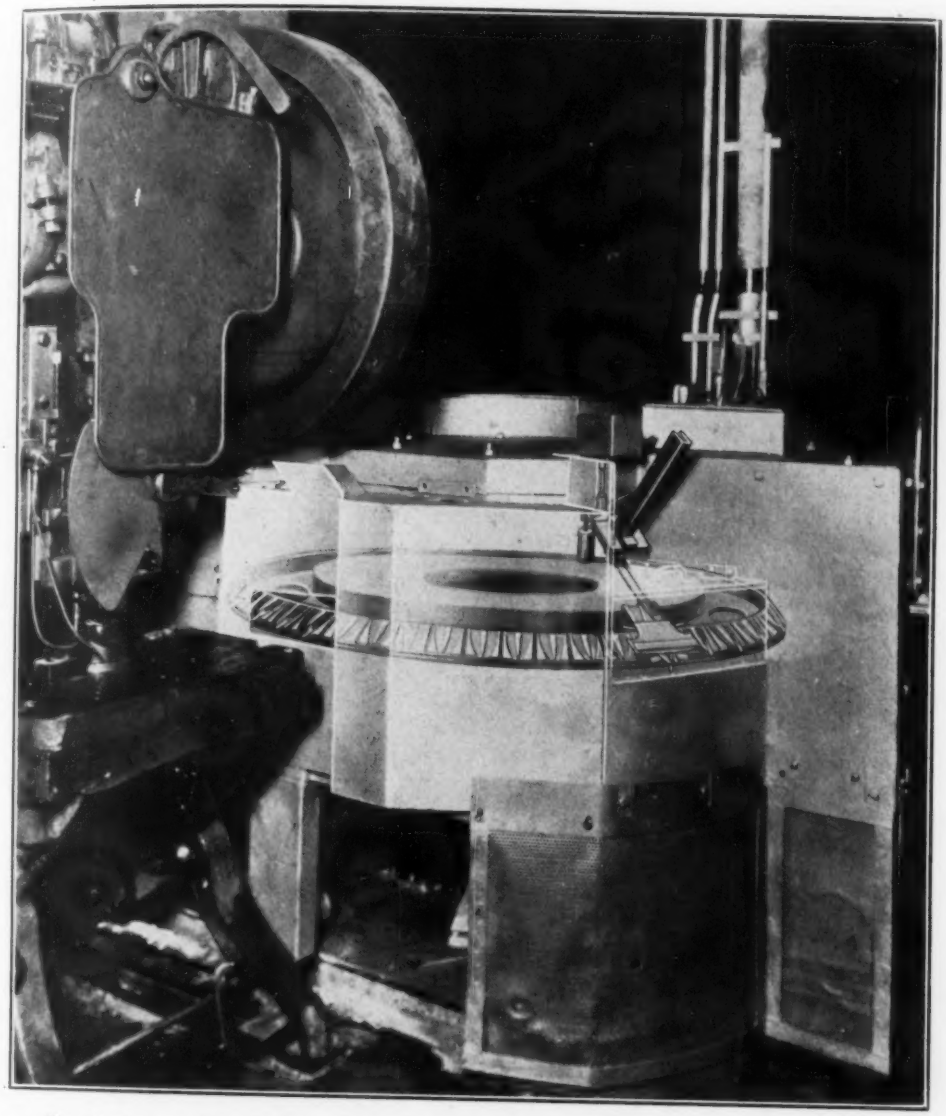


Fig. 6—Rotary Hearth Furnace for Heating Handset Receiver Cores Previous to Hot Forming.

efficiency is obtained by the use of the recuperator used in conjunction with this furnace. The exhaust gases from the furnace heat the incoming air in the recuperator to 650 degrees Fahr. These billets are heated to 1800 degrees Fahr. with an allowable temperature variation of ± 15 degrees Fahr.

Rotary Hearth Furnace for Hot Forming Receiver Cores

The magnetic cores used in the receivers of the new handset

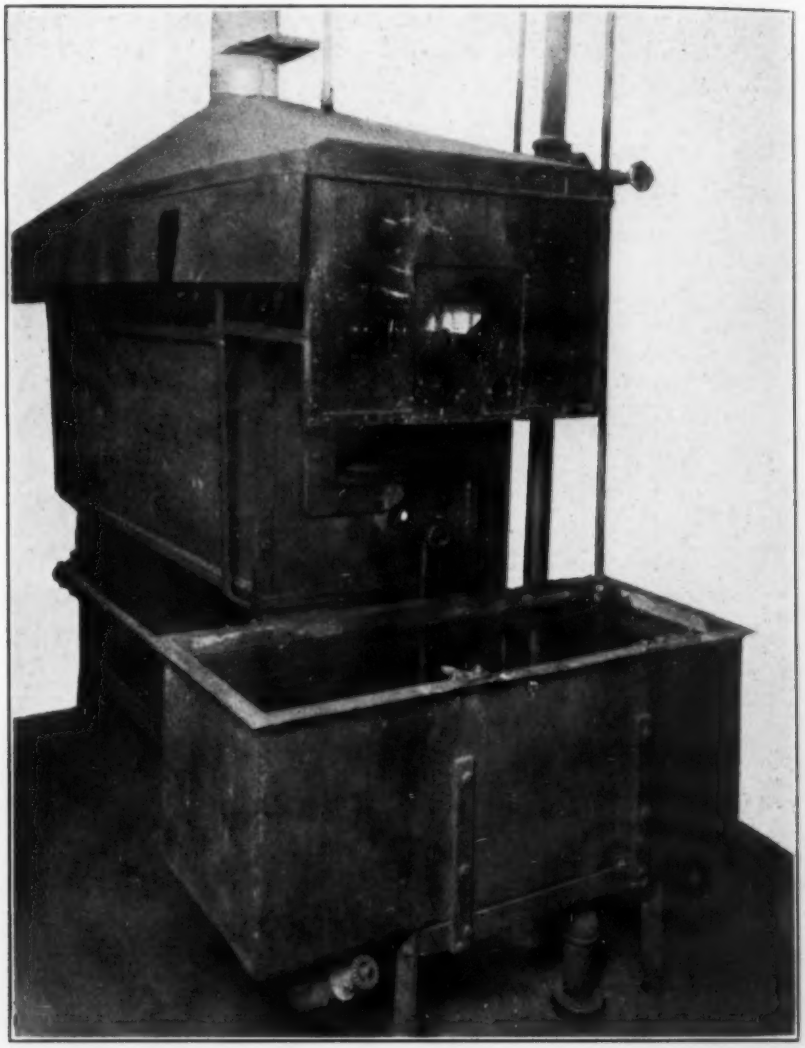


Fig. 7—Shows the Furnace Formerly Used for the Hardening of Magnets. This was an Oil Fired Furnace.

telephone instrument are silicon steel punchings which have an irregular shape and are about one inch long and a maximum of one-half inch wide. These stampings are bent at right angles in the process of manufacture. To prevent the cracking of this relatively brittle material during this forming operation, it is necessary to form the parts hot.

Until recently these parts were heated in a gas-fired furnace, shown in Fig. 5. A number of the parts were laid on an iron bar which was placed in the furnace, and brought to a temperature above a red heat. When heated, the bar was removed from the furnace by

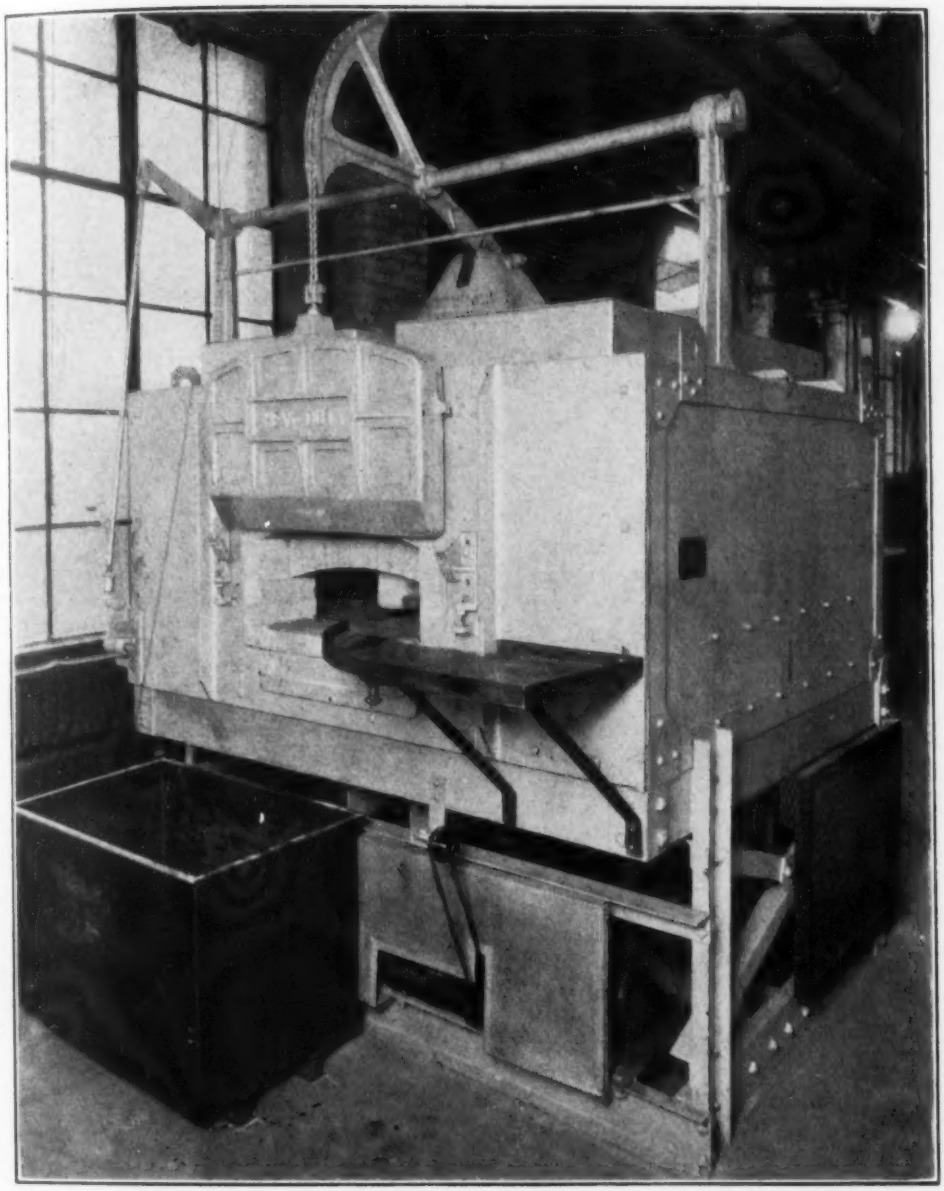


Fig. 8—Rotary Hearth Electric Magnetic Hardening Furnace which Replaced the one Shown in Fig. 7.

an operator and placed in proximity to the forming punch press where another operator removed the parts separately and placed them in the forming die. The actual forming temperatures of the parts were obviously not uniform as the last parts removed from the rapidly cooling bar were somewhat too cold for satisfactory forming. This method of heating parts has been replaced by a continuous

process by which all of the parts to be formed are at the proper forming temperatures when placed in the forming tool.

This improved type of furnace, shown in Fig. 6, is of the rotary hearth type. The receiver cores to be formed are placed in the magazine shown extending from the top of the furnace at the right side. By means of a solenoid operated trip the parts are slid into slots in

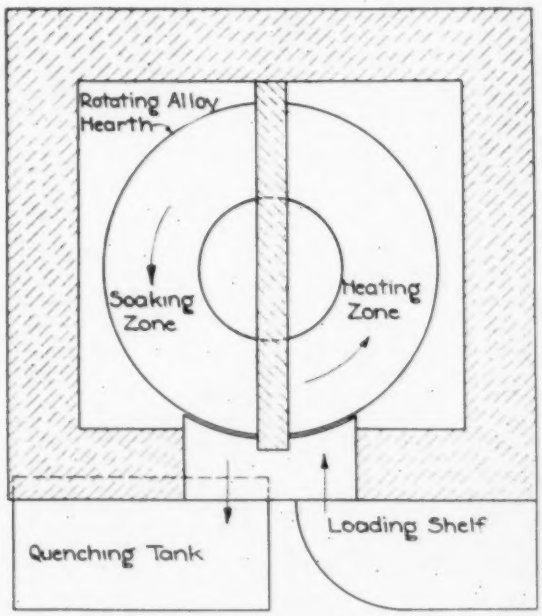


Fig. 9—A Section of the Furnace in Plan View, Shown in Fig. 8.

the rotating hearth. The temperature of the furnace and the speed of the hearth are so controlled that the part attains the predetermined forming temperature by the time it reaches the opening at the side of the furnace adjacent to the forming press. While it might appear that it would be uncomfortable for the press operator to work so close to this furnace, this condition has been taken care of by cooling the side of the furnace directly in front of the discharge opening with a blast of cold air blown between the outer layer of heat insulation and the outer steel shell of the furnace. At this point the press operator picks the part from the slot in the hearth with a pair of long nose pliers and places it into the forming die. This principle of operation and the construction of the magazine and hearth is illustrated by Fig. 6.

This furnace and press installation has insured that the receiver

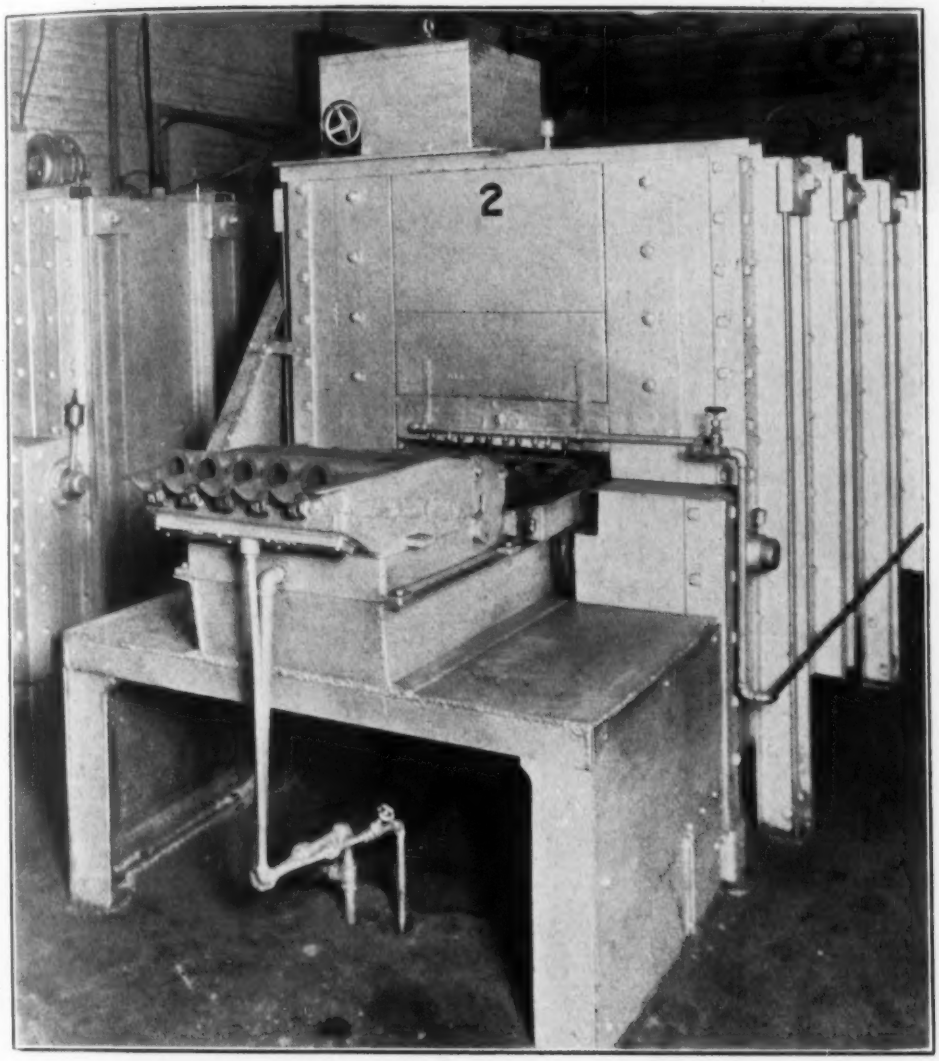


Fig. 10—Automatic Core Ring Stacker for Continuous Annealing Furnace.

cores are formed at close temperature limits which has materially improved the magnetic uniformity of these piece parts which play such an important part in the satisfactory operation of the telephone.

Rotary Hearth Electric Magnetic Hardening Furnace

Until two years ago the hardening of magnets was carried on in a small double end oil-fired furnace shown in Fig. 7. The magnets were charged in one end of this furnace and pushed or pulled through with a hook by the operator to the discharge end where they were removed and quenched in tongs. This necessitated frequent trips

of the operator to the opposite end of the furnace. The capacity of this furnace was only about 90 pounds per hour and the manual temperature control made the quality of the output somewhat uncertain.

This furnace was replaced by an electric furnace shown in Fig. 8. The outside overall dimensions of the furnace are approximately 7 feet, width 6½ feet, height 7 feet not including the door raising mechanism. The radiation losses at 1800 degrees Fahr. resulting

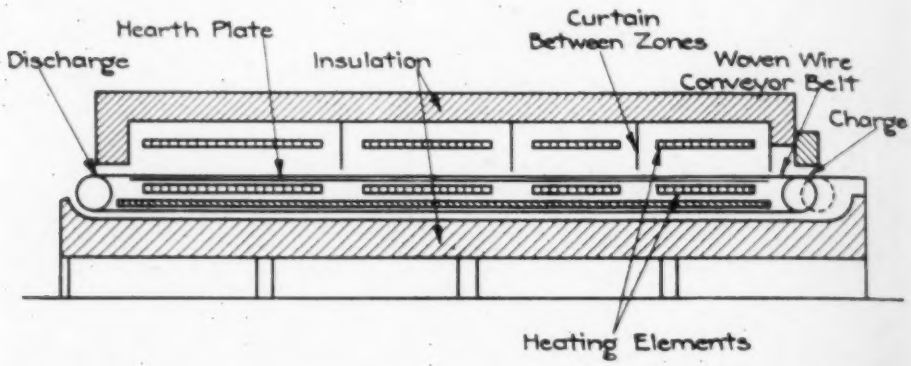


Fig. 11—Shows in Simplified Form the Construction of the Furnace Shown in Fig. 10.

from this construction do not exceed 13 kilowatts. The furnace heating elements are located in the arch, sidewalls, floor and front and back walls of the furnace. Their distribution in the furnace was carefully planned so that close temperature uniformity of the product leaving the discharge door of the furnace can be realized. Power is supplied to this furnace by two 30 K.W., 3-phase, 440-volt primary, 110-volt secondary, 60 cycle transformers. These transformers are located beneath the furnace shell.

The magnets to be hardened are fed by the operator from the loading shelf at the right side of the furnace door upon a rotating hearth. This rotating hearth is made of cast heat resisting metal and is of such design that warpage is reduced to a minimum. This hearth is rotated in a counter clockwise direction by means of a central vertical shaft which is supported by a bearing under the furnace shell. The rotation of the hearth can be varied from a minimum speed of one revolution in twenty minutes to a maximum speed of one revolution in five minutes.

A section of the furnace in plain view is shown in Fig. 9. The heating chamber is divided into two zones above the rotating hearth

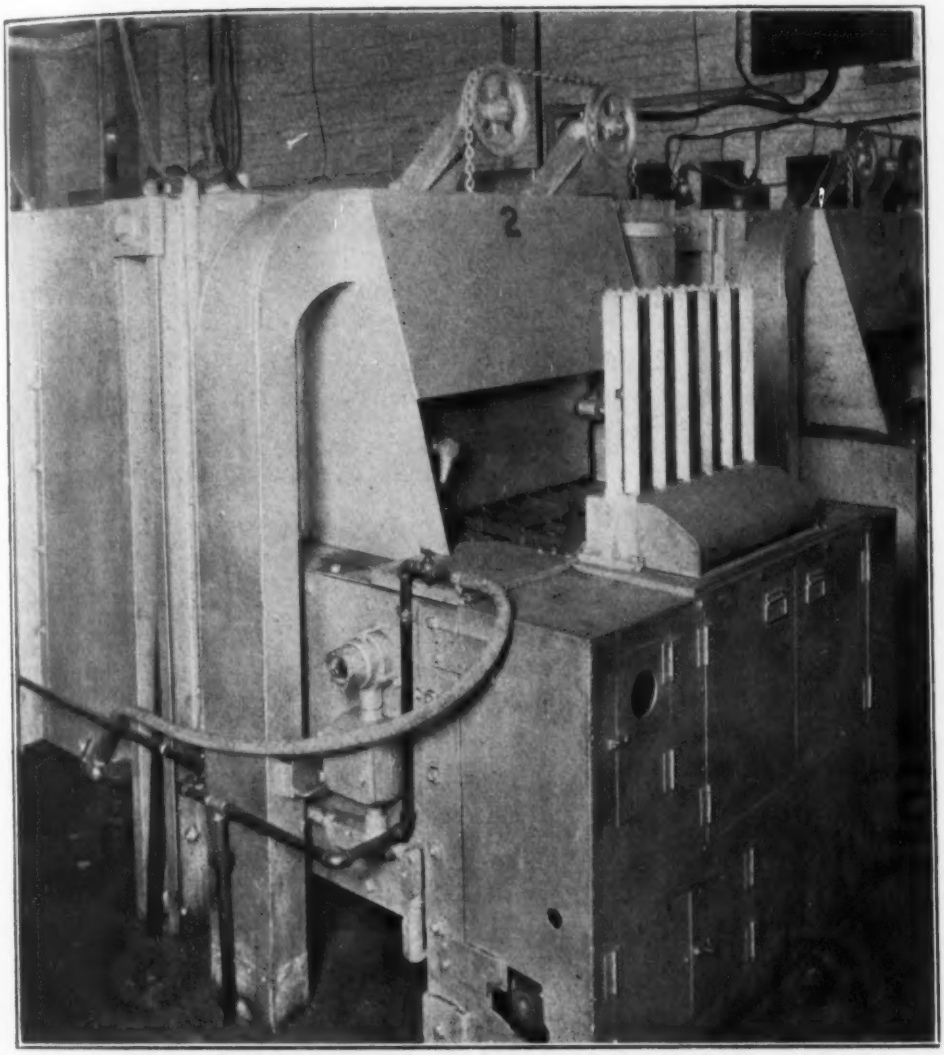


Fig. 12—Automatic Core Ring Loader for Continuous Annealing Furnace.

by a hanging refractory wall. These zones are called the heating and soaking zones. The purpose of this partition is to permit the heating and soaking zones to be operated at somewhat different temperatures. The speed of rotation of the hearth is so controlled that the parts placed upon it by the operator come up to temperature before passing under the dividing partition into the soaking zone. The magnets remain in the soaking zone until they have attained the predetermined temperature throughout. When a magnet again reaches a point at the left side of the furnace door, the operator picks it up with a pair of tongs, quickly draws it out of the furnace and quenches

it in the water tank in front of the furnace. Hand quenching with tongs has been found necessary to prevent warping of certain formed parts during this operation.

The heating and the soaking zones are separate electric circuits and the temperatures of each of these zones are automatically controlled by a recording controller. By regulating the zone temperatures and by changing the speed of the rotating hearth, magnets of various weights and shape can be satisfactorily hardened. The furnace and auxiliary temperature control equipment is so designed that it can deliver 300 pounds of magnets per hour at a temperature of 1800 degrees Fahr. with a temperature variation of plus or minus 15 degrees Fahr.

Permalloy Dust Core Ring Heat Treating Furnace

The furnace shown in Fig. 10 is an electrically heated furnace for the heat treatment of permalloy dust core rings. These core rings are used in particular for the magnetic cores of loading coils which are placed in telephone circuits to improve transmission. These rings are made from the nickel-iron alloy permalloy which had been pulverized to a fine powder and subsequently molded under high pressure to form rings of the required size and thickness. Most of the core rings manufactured have the following dimensions, outside diameter 3 inches, inside diameter 2 inches, thickness ¼ inch. In order to bring out the desired magnetic characteristics in the core rings a very precise and accurate heat treatment is required.

The general heat treating procedure is to place the core rings on a moving belt at the supply end of the furnace. This belt carries them through the furnace at a definite and predetermined speed, enabling them to pass through the specified time-temperature cycle before they emerge at the discharge end of the furnace. The heat treatment for these core rings consists in holding them at approximately 1000 degrees Fahr. for three minutes and in seeing that the time required to bring the cores to this temperature does not exceed ten and one-half minutes.

The overall dimensions of the furnace are 25 feet long, 5 feet 4 inches wide, and 6 feet high. The dimensions of the heating chamber are 18 feet long, 24 inches wide, and 20 inches high.

Fig. 11 shows in simplified form the construction of the furnace. The conveyor belt which carries the core rings through the

furnace is made of woven heat resisting alloy wire. This belt passes over cast iron-chromium drums at either end of the furnace. Proper belt tension is maintained by means of counterweights at the supply end of the furnace. This conveyor belt is supported on channel shaped alloy hearth plates which are continuous throughout the length of the chamber. This conveyor belt is driven at a normal speed of 86 feet per hour. Maximum and minimum speeds of 27 and 108 feet per hour, respectively, are obtainable.

The heating of the furnace is accomplished in four zones each having a separate set of heating elements and controlled separately. The heating elements of each of these zones are distributed among three phases of power input. The elements of the first zone consist of one phase suspended from the arch and two phases under the hearth. The top and bottom phases when connected in Y connection provide a maximum connected load of 45 K.W., on a 3-phase, 60-cycle, and 110-volt circuit. The elements of the second, third and fourth zones are similarly located and connected and amount to total connected loads of 36 K.W., 30 K.W. and 30 K.W., respectively.

Power is supplied to the furnace through one 150 K.V.A., 3-phase, air-cooled transformer operating on 60 cycles and wound for 440-volt primary and 110-volt secondary.

To accomplish the heat treating cycle previously mentioned the temperature controlling equipment for each zone is set at a temperature value previously determined by test to be the proper for this process. The belt is started and the power is turned on the furnace and controllers. The speed of the belt is adjusted and maintained such that the time required for a core part to pass through the furnace is sufficient to insure satisfactory heat treatment of the core part.

Operators are required only for maintaining an adequate supply of rings to load the feeding mechanism shown in Fig. 12 and for removing rings from the stacking device shown in Fig. 10.

Conclusion

The furnaces which have just been described are typical of the equipment used by the Western Electric Company to insure that the heat treatment of the parts entering into its apparatus is uniform and that the parts produced meet, in every respect, the very exacting re-

quirements placed upon them. That the effort expended upon the selection and development of improved heat treating equipment has been fully justified has been demonstrated by the progress made in manufacturing improved and more efficient telephone equipment.

DISCUSSION

Written Discussion: By C. N. Stevenson, district Manager, Hevi-Duty Electric Co., Milwaukee.

The use of small automatic continuous furnaces described in Mr. Timm's paper is one of the interesting developments in furnace design in the last few years. Large production furnaces are quite generally equipped with automatic labor saving devices, but when a small part is to be heated too little thought is given to the possibility of obtaining an automatic heating machine built to fit the particular job. More likely than not a small standard box type furnace is chosen from a catalogue and the development work on that particular job is finished.

The furnace manufacturers are probably to blame for this condition for, like other equipment manufacturers, they have tried to standardize their products.

But special automatic furnaces for small work show greater proportionate returns than automatic equipment on large furnaces. The principal cost of a furnace is the initial cost, the fuel or electric consumption and the labor cost. On small furnaces the labor cost is by far the largest item. On such applications the elimination of an operator or the increased output per operator will pay for the furnace in a very short time. Certainly the additional expense of one or two thousand dollars for automatic handling to save one operator has greater justification than five or ten thousand dollars for the same purpose on a large installation.

From the furnace manufacturer's viewpoint, the building of special furnaces is not as difficult as it was a few years ago. The past several years have brought out small units which are well adapted to the automatic continuous method and these furnaces have almost become considered as standard equipment. The small rotary hearth, mesh conveyor, small pusher and sliding rail furnaces are in this class. Manufacturers have adapted their standard refractories, elements, patterns etc., to these types so that delivery and maintenance is no longer a problem. Experience gained in building and operating these types of furnaces makes it possible to build them of any size or design and be reasonably certain of what to expect in the way of temperature distribution and production.

As Mr. Timm states in his paper, there are jobs similar in all plants which require standard box type furnaces, but for production work, even for small applications, building the furnace to fit the particular job is preferable to attempting to make the job fit a standard piece of equipment.

ESTIMATION OF INTERNAL STRESS IN QUENCHED HOLLOW CYLINDERS OF CARBON TOOL STEEL

By O. V. Greene

Abstract

The internal stresses in hollow cylinders of carbon tool steel quenched by two widely different methods have been determined. The stresses at the holes of hollow cylinders quenched by submerged flushing are either tangential tension, or a combination of tangential tension and compression. The stresses at the holes of cylinders quenched by flushing at the holes were uniformly tangential compression. The latter treatment in using the quenching stresses to advantage adapts such cylinders to better withstand internal pressures. The axial stresses at holes of all cylinders when present were compressional. However the values of the axial stresses were small and therefore of little consequence.

Methods for determining internal stresses and complete derivation of the formulas used for the estimation of tangential and radial stresses are included.

THE mechanism of stress formation during quenching has received able treatment from several investigators, notably, Hoyt[1], Scott[2], and Heyn[3]. However, there is little information in the literature on the actual magnitudes or distribution of the residual stress so formed. It is a matter of common knowledge that quenching sets up stresses that may cause cracking or warping. In spite of this general knowledge and the importance of the subject, there is almost a complete lack of specific information on this phase of the treatment of metals.

Industry is demanding more and more of its tools every day.

[1]S. L. Hoyt, "Stresses in Quenched and Tempered Steel," Transactions, American Society for Steel Treating, Vol. XI, 1927, p. 509.

[2]H. Scott, "Origin of Quenching Cracks," Scientific Paper of the Bureau of Standards, No. 513.

[3]E. Heyn, "Physical Metallography," translated by M. A. Grossmann, John Wiley & Sons, 1925.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, O. V. Greene, a member of the society, is connected with the metallurgical department of the Carpenter Steel Co., Reading, Pa. Manuscript received May 29, 1930.

Table I
Analyses of Steels

Steel No.	Per Cent C	Mn	Si	P	S	Cr	Ni
S	0.89	0.30	0.12	0.013	0.016	0.08	0.07
M	0.91	0.25	0.18	0.013	0.016	0.07	0.10
D	0.92	0.30	0.20	0.016	0.010	0.08	0.10

Substantial increases in production are only possible with better tools. Alloy steels met the demand until a short time ago, but now are being taxed to the limit. Might not scientific quenching offer untold possibilities? If there exist forces that are sufficient to cause complete failure, why not use them to advantage instead of allowing them to defeat the very purpose of heat treatment.

Methods of Stress Determination

However before such a method of heat treatment can be planned, it is necessary to consider methods by which the internal stresses formed in quenching can be evaluated. A complete method for the determination of axial stresses in solid specimens has been developed by Heyn (See Appendix I). The results of the application of this method to three carbon tool steels of approximately the same analysis, but with widely different hardness penetrations, is given in Table II. Only the maximum stresses exhibited by each specimen in a surface layer of 3/64 inch have been given. The analyses of these steels are given in Table I, and the hardness penetrations of specimens ¾ inch in diameter quenched in brine from the temperatures indicated, in Fig. 1. It is interesting to note that the axial stress decreases as the hardness penetration increases. The stress in any one steel is seen to increase with quenching temperature, with the exception of steel D at 1550 and 1600 degrees Fahr. where the specimens nearly or completely harden through respectively.

The present paper deals with the development of a method for the estimation of internal stress in quenched hollow cylinders, and its application to two distinct types of quenching. Hollow cylinders were chosen since they represent, for instance, the ideal forms of drawing or cold heading dies, in which the most important stresses are tangential and radial, rather than axial. A complete development of the method by which the magnitude, nature and distribution of such stresses may be determined is given in Appendix II. It is suf-

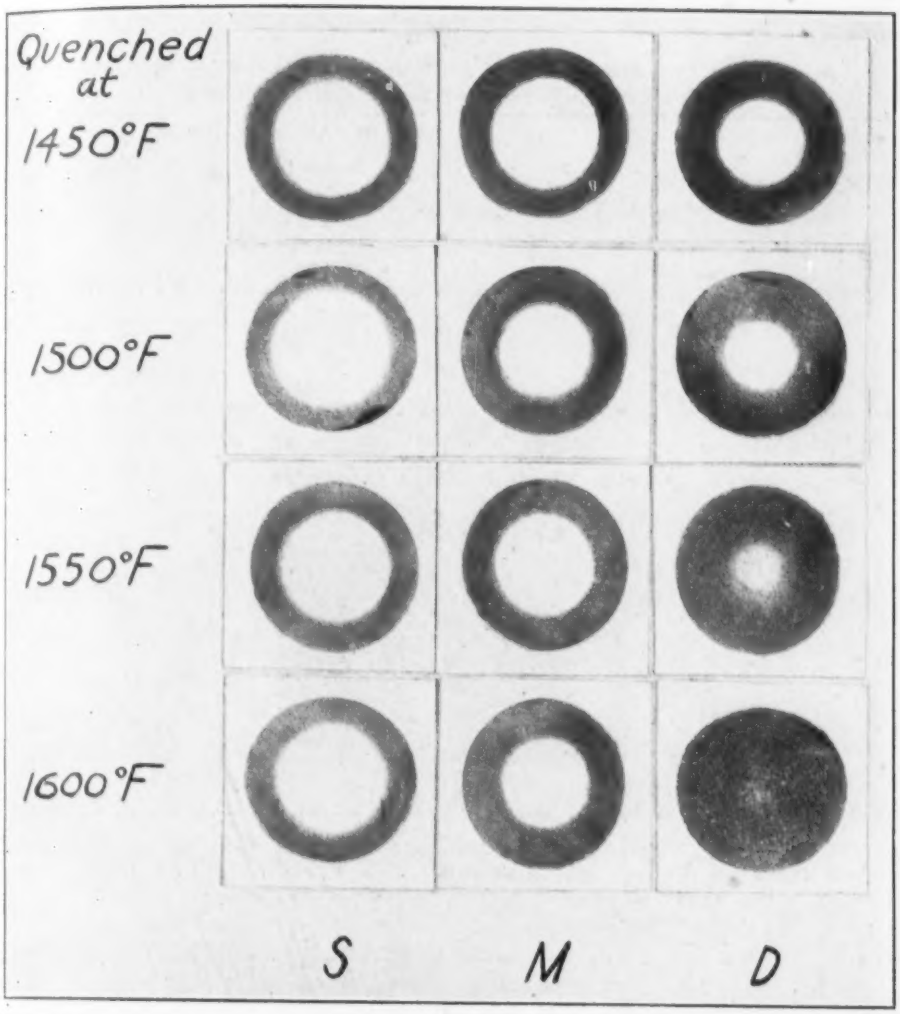


Fig. 1—Hardness Penetrations of Three Carbon Tool Steels ¾ Inch in Diameter Quenched in Brine at the Temperatures Indicated. Actual Size.

ficient at this point to mention only briefly the method used. Before proceeding, however, the premise on which Heyn's method and the development here is based, must be understood. This simple law states that all iron base alloys have the same elastic properties irrespective of composition or heat treatment. For example, low carbon, high carbon or alloy steels all have a modulus of elasticity of 30,000,000, whether they be in an annealed or hardened condition. Consequently if the application or removal of stress results in a unit deformation of 0.001 inch, this always corresponds to an application or release of 30,000 pounds per square inch. Therefore, if layers be removed from the outside or inside diameter of hollow

Table II

Axial Stress in Surface Layer of 3/64" in Bars 3" Long ¾" in Diameter Quenched in Brine from Temperatures Indicated

Quenching Temp. Degrees Fahr.	Axial Stress, Lbs. per Sq. In.		
	Steel S	Steel M	Steel D
1450	—175,000	— 94,000	—119,500
1500	—172,000	—135,600	—123,400
1550	—188,300	—146,000	—102,600
1600	—182,500	—173,200	— 97,000

Note: In this and following tables showing stress, the minus sign denotes compression and the plus sign tension.

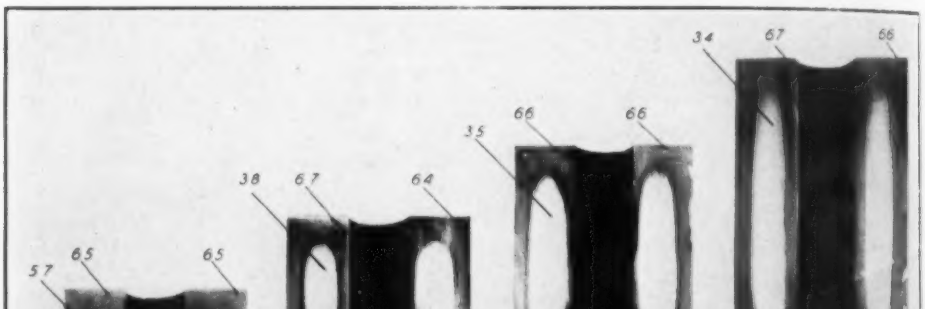


Fig. 2—Cylinders Quenched by Submerged Flushing at 1600 Degrees Fahr. Rockwell C Hardness Numbers are as Shown. Note Cylinder ¾ Inch Long Hardened Through at this Temperature.

cylinders, and the change in inside or outside diameter respectively be measured it is possible to evaluate the tangential and radial stresses released. The corresponding stresses in the layers removed when they were part of the original cylinder may also be obtained. Axial stresses may be determined by observing the length changes as successive layers are removed.

Quenching Methods

The cylinders used in the following work were made of carbon tool steel of the type M, shown in Fig. 1. All specimens were 1⅞ inches in diameter with a ⅝-inch hole; the lengths were varied in multiples of ¾ inches, giving cylinders ¾, 1½, 2¼ and 3 inches long.

A number of cylinders of each length were quenched by submerged flushing at various temperatures. Certain specimens were

Table III

Rockwell C Hardness

Length	Submerged Flush Quenched at 1600° F.	Submerged Flush Tempered 450° F.	Flushed at Hole Quenched at 1600° F.	Flushed at Hole Tempered 450° F.
	57	52	37	36
¾"	65	60	38	36
	65	61	65	60
			67	60
	38	36	34	34
1½"	67	60	36	35
	64	59	66	60
			66	61
	35	35	34	33
2¼"	66	60	34	34
	66	60	66	61
			67	60
	34	34	34	34
3"	67	60	35	35
	66	59	66	60
			66	59

Note: Hardness values at points indicated in Figs. 2 and 5.

subsequently tempered. This type of heat treatment followed by tempering resembles one that is commonly used for header dies. During this work, few failures of cylinders occurred during quenching. The characteristic hardness penetrations after flushing at 1600 degrees Fahr. are shown in Fig. 2. It is interesting to note that the cylinder ¾ inches long hardened through.

The balance of the specimens were flushed at the hole only, as suggested by L. S. Cope[4]. The first attempts with this type of quenching on cylinders over ¾ inches long resulted in many failures. Cylinders after heating to the required temperature were placed in a crude device consisting merely of a recessed steel block and a nozzle with the head fitting over the top of the specimen. As flushing proceeded, there was considerable spilling which caused unequal and irregular hardness penetrations. This condition gave rise to unbalanced stresses which caused cracking of many cylinders before they were removed from the flushing device. Fig. 3 shows the results of these tremendous unbalanced forces and the nonuniform hardness penetrations obtained. The stresses causing the failures were, of course, surface tangential stresses of high order. A conception of the forces involved may be realized when it is seen that they caused complete failure of a 3-inch cylinder with a ⅝-inch wall.

[4]L. S. Cope, "Hardening Cold Heading Dies," TRANSACTIONS, American Society for Steel Treating, Vol. XIV, 1928, p. 51.

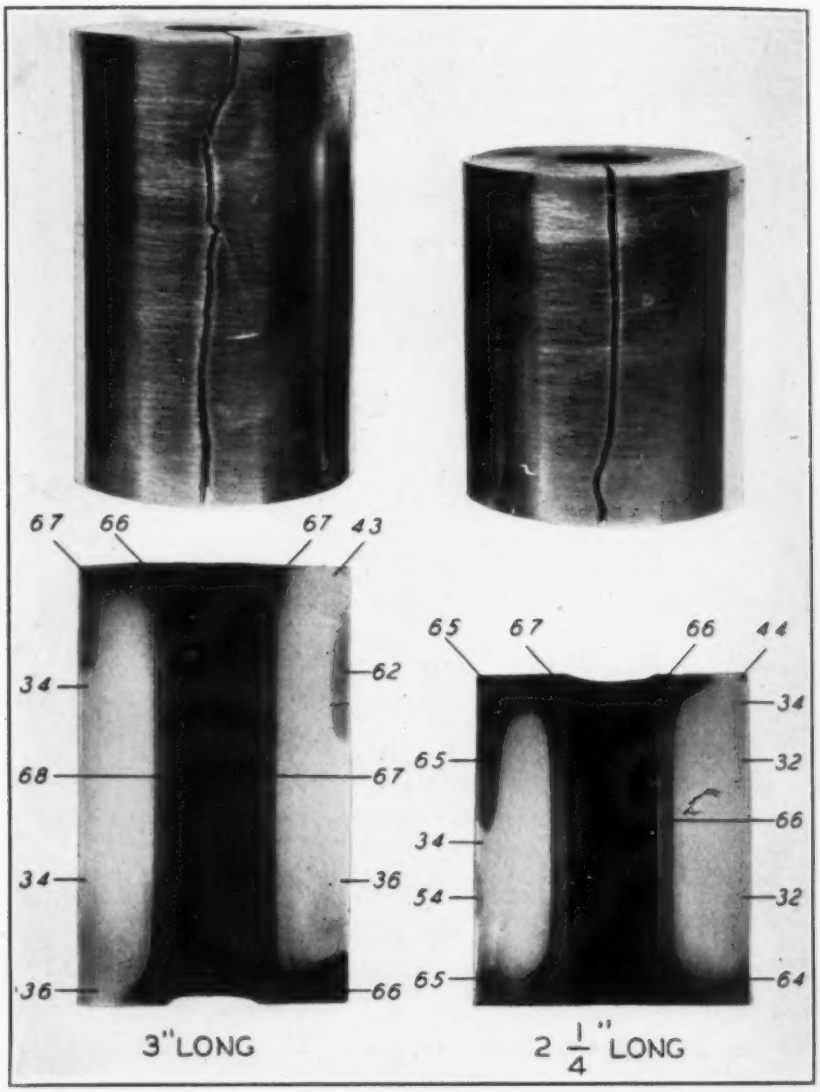


Fig. 3—Results of First Attempts to Flush the Hole Only. The Cylinders Failed by Complete Cracking when Room Temperature was Reached. The Lower Half of the Figure Shows the Nonuniform Hardness Penetration Obtained and the Rockwell C Hardness Numbers. Flushed at Hole 1600 Degrees Fahr.

The quenching difficulties encountered in flushing the hole were entirely overcome by the use of a simple quenching device shown in Fig. 4. Two disks of stainless steel ⅜ inches thick and 4 inches in diameter were drilled as shown. The bar holding the spreader was peened in the bottom disk. Asbestos washers were placed between the cylinders and disks, and the whole clamped by means of stainless steel bolts and nuts. The holes in the disks corresponding to the holes in the cylinders were tapered to admit the tapered nozzle

Table IV
Rockwell C Hardness

	Hardness gradation in cylinders 3" long (flushed at hole) taken at intervals of $\frac{1}{32}$" from inside to outside surface on the center of cross section	
	Quenched at 1450° F.	Quenched at 1600° F.
Inside surface	66	66
	58	61
	45	55
	43	49
	39	44
	37	40
	36	38
	36	36
Outside surface	35	36

of the flush at the top and to allow unrestricted flow at the bottom. The disks and cylinders after assembly were placed in the furnace and heated to the required temperature. The quenching operation was consequently extremely simple. The device containing the cylinder was removed from the furnace, the nozzle of the flush inserted and the water turned on. The fit between disks and cylinders prevented the flushing of the top or bottom surfaces. The alignment of cylinders with respect to the spreaders was always perfect. The extreme uniformity of results obtained with this device is shown in Fig. 5.

Hardness Tests

The C Rockwell hardness obtained by both methods of quenching have been incorporated in Figs. 3 and 5. The hardness values at the same points after tempering at 450 degrees Fahr. are given in Table III. The gradation in hardness from the hardened zone outward, in cylinders flushed at the hole at 1450 degrees Fahr., as compared to cylinders flushed at the hole at 1600 degrees Fahr., is given in Table IV. The drop in hardness over the cross section of the cylinder flushed at 1600 degrees Fahr. is seen to be much less than the cylinder flushed at 1450 degrees Fahr. This is undoubtedly one reason why the hardened surfaces of dies that have been flushed at 1600 degrees Fahr. do not tend to sink as readily as those of dies quenched at lower temperatures.

Dimensional Changes on Quenching

The measurements for the determination of dimensional changes were taken on a number of cylinders at the points shown in Fig. 6.

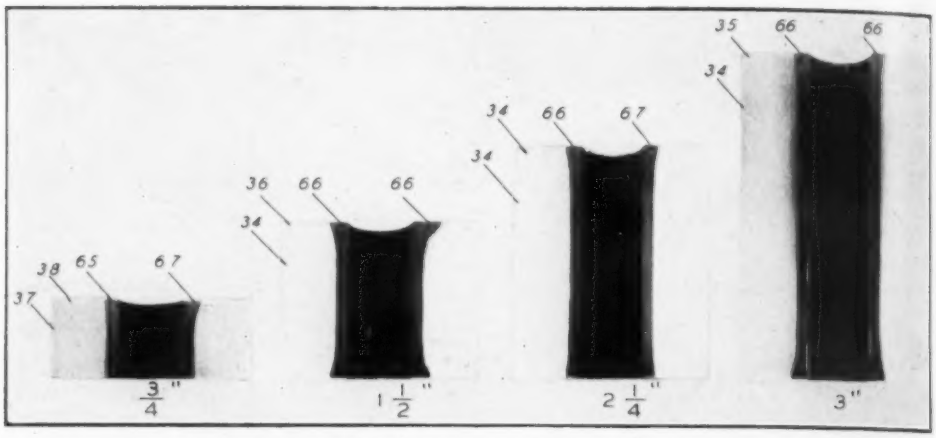


Fig. 5—Cylinders Quenched by Flushing the Hole at 1600 Degrees Fahr. Rockwell C Hardnesses at Points Shown. Note the Uniform Hardness Penetration as Compared to Fig. 3.

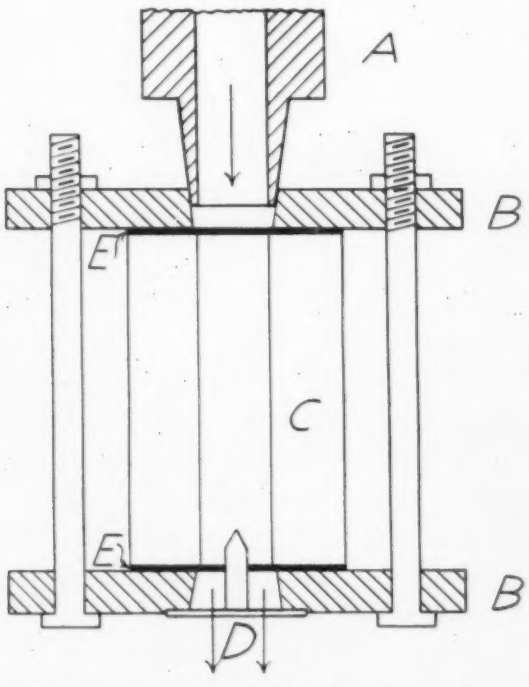


Fig. 4—Quenching Device for Flushing the Holes of Cylinders that Resulted in Uniform Hardness Penetrations. A—Nozzle of Quenching Fixture; B—Stainless Steel Disks, Bolts and Nuts; C—The Cylinder in Position for Flushing; D—Spreader; E—Asbestos Packing Rings.

The total change at each of these points after submerged flushing at 1450 degrees Fahr., and flushing at the hole at 1450 degrees Fahr., is given in Table V. The changes after the same methods of quenching, but at 1600 degrees Fahr. are given in Table VI. The infor-

Table V

Dimensional Change

Cylinders Quenched at 1450° F.

Length of Cylinder Inches	Diameter Change of Hole Inches			Outside Diameter Change Inches			Length Change Inches		
	A	B	C	D	E	F	G	H	I
				Submerged Flush as Quenched					
¾	+.0016	.0000	+.0010	+.0015	−.0004	+.0005	+.0012	+.0023	+.0033
1½	+.0020	−.0040	+.00105	+.0024	+.0009	+.0012	−.0010	−.0020	+.0012
2¼	+.00305	−.0031	+.0038	+.0030	−.0010	+.0020	+.0008	−.0006	+.0013
3	+.00195	−.0035	+.0018	+.0015	−.0008	+.0020	−.0024	−.0010	+.0003
				Submerged Flush—Tempered at 450° F.					
¾	+.0001	−.0019	+.00105	+.0018	−.0009	.0000	+.0009	+.0029	+.0042
1½	+.0005	−.0055	+.0003	+.0021	+.0015	+.0011	+.0004	−.0010	+.0023
2¼	+.0011	−.0042	+.0020	+.0008	−.0014	+.0005	+.0005	−.0009	+.0005
3	+.0015	−.0048	+.0010	+.0001	+.0002	+.0005	−.0030	−.0020	−.0004
				Flushed at the Hole as Quenched					
¾	−.0008	−.0004	−.0012	+.0010	+.0005	+.0013	+.0003	.0000	+.0031
1½	−.0012	−.0022	−.0010	+.0015	+.0008	+.0010	−.0018	−.0011	+.0020
2¼	−.0005	−.0011	−.0005	+.0004	+.0002	+.0006	−.0021	−.0019	+.0005
3	−.0011	−.0009	−.0010	+.0010	+.0011	+.0018	−.0025	−.0012	+.0002
				Flushed at the Hole, Tempered at 450° F.					
¾	−.0004	−.0002	−.0008	+.0005	+.0005	+.0006	−.0003	+.0005	+.0035
1½	−.0024	−.0032	−.0020	+.0006	+.0009	+.0009	−.0018	−.0010	+.0030
2¼	−.0014	−.0020	−.0020	−.0004	−.0008	−.0002	−.0021	−.0021	−.0003
3	−.0020	−.0018	−.0024	+.0005	+.0004	+.0008	−.0028	−.0026	−.0008

mation in these tables is the average of at least three specimens for each length and treatment. The dimensional changes after submerged flushing at 1600 degrees Fahr., as compared to flushing at the hole at 1600 degrees Fahr., are shown much exaggerated in Fig. 7.

These results show that all lengths of cylinders, both at 1450 and 1600 degrees Fahr., after submerged flushing, are bell-mouthed. That is, the middle of the hole decreased in size while the ends expanded. The distortion is greater at 1600 degrees Fahr., than at 1450 degrees Fahr. After tempering at 450 degrees Fahr., the bell form persisted, but both the ends of the hole and the middle showed a considerable decrease in diameter. Flushing at the hole presented entirely different results. The holes are seen to be but slightly warped, and both the ends and the middle of the holes show a uniform decrease in diameter with all lengths at both 1450 and 1600 degrees Fahr. At 1450 degrees Fahr., the holes of the ¾ inch and the 3 inch, and at 1600 degrees Fahr. the holes of the 1½ and 2¼ inch cylinders, actually showed less decrease in diameter at the middle than at the ends. This caused a slight warping in a direction opposite that of the cylinders quenched by submerged flushing. The cylinders flushed at 1600 degrees Fahr. show a greater decrease in hole diameter than the cylinders flushed at 1450 degrees Fahr. Tem-

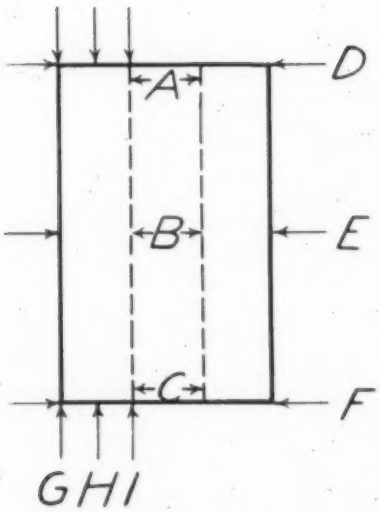


Fig. 6—Diagram Showing Points on Cylinders at which Readings Were Taken for the Determination of the Size Changes.

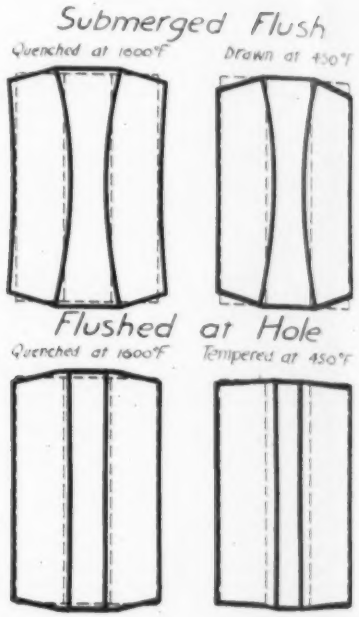


Fig. 7—Dimensional Changes of Cylinders 3 Inches Long After Quenching at 1600 Degrees Fahr. by Submerged Flushing and Flushing at the Hole, and After Tempering at 450 Degrees Fahr. Much Exaggerated.

pering at 450 degrees Fahr. further decreased the diameter of the holes.

There was considerably more bending at the outside diameters of cylinders quenched by submerged flushing than those quenched by flushing at the hole. Tempering at 450 degrees Fahr., however, reduced the bending after both methods of quenching at both temperatures.

The length changes of all specimens were considerably greater at the hole, causing a distinct crowning at each end. These total changes increase slightly with increasing quenching temperatures with both methods of quenching. However, the length changes after quenching by both methods at 1450 and 1600 degrees Fahr. decrease with increasing length of cylinder. After tempering at 450 degrees Fahr., the lengths of all cylinders decreased with the exception of the ¾ and 1½ inch cylinders flushed at the hole, and quenched by submerged flushing at 1450 degrees Fahr. Both of these lengths after tempering showed substantial increases.

Table VI
Dimensional Change
Cylinders Quenched at 1600° F.

Length of Cylinder Inches	Diameter Change of Hole Inches			Outside Diameter Change Inches			Length Change Inches		
	A	B	C	D	E	F	G	H	I
Submerged Flush as Quenched									
¾	+.00228	−.0011	+.0028	+.0046	+.0012	+.0033	+.0019	+.0027	+.0042
1½	+.0023	−.0053	+.0025	+.0030	+.0008	+.0020	−.0004	−.0012	+.0028
2¼	+.0048	−.0054	+.0052	+.0041	+.0015	+.0040	−.0016	−.0012	+.0025
3	+.0035	−.0031	+.0030	+.0016	+.0005	+.0017	−.0005	.0000	+.0020
Submerged Flush, Tempered at 450° F.									
¾	+.0020	−.0016	+.0024	+.0047	+.0018	+.0037	+.0012	+.0021	+.0038
1½	+.0012	−.0063	+.0012	+.0031	+.0017	+.0025	−.0009	−.0016	+.0020
2¼	+.0040	−.0064	+.0044	+.0020	.0000	+.0023	−.0022	−.0018	+.0015
3	+.0025	−.0042	+.0024	+.0005	+.0003	+.0008	−.0020	−.0010	+.0007
Flushed at Hole as Quenched									
¾	−.0017	−.0016	−.0015	+.0012	−.0006	+.0010	+.0002	+.0004	+.0033
1½	−.0020	−.0018	−.0018	+.0002	−.0008	−.0001	−.0008	−.0016	+.0030
2¼	−.0022	−.0016	−.0019	+.0010	+.0009	+.0011	−.0007	−.0005	+.0025
3	−.00265	−.0030	−.0024	+.0002	−.0001	+.0004	−.0003	+.0004	+.0021
Flushed at Hole, Tempered at 450° F.									
¾	−.0032	−.0035	−.0030	+.0011	−.0006	+.0009	+.0001	+.0002	+.0020
1½	−.0037	−.0027	−.0038	+.0008	.0000	+.0002	−.0009	−.0016	+.0018
2¼	−.0033	−.0031	−.0039	+.0010	+.0008	+.0009	−.0004	−.0005	+.0012
3	−.0040	−.0045	−.0041	+.0006	.0000	+.0003	−.0001	−.0004	+.0008

Table VII
Distribution of Stress in Cylinders ¾" Long

Layer No.	$(r_o - r_n)$ Inches	Tangential Stress Lbs. Per Sq. In.	Radial Stress Lbs. Per Sq. In.	Tangential Stress in Action with 100,000 Lbs. Per Sq. In. at Hole Lbs. Per Sq. In.
Cylinder No. 2 Flushed at Hole at 1450° F.				
Outside Surface 1	.0000	+120,600	−40,300	+145,700
2	.0446	+ 65,200	−33,190	+ 91,000
3	.0915	+ 13,210	−14,780	+ 41,810
4	.1410	+ 6,555	−14,457	+ 37,255
5	.1886	+ 54,370	−30,428	+ 87,270
6	.2372	+ 68,385	−38,775	+104,385
7	.2864	+ 25,200	−30,641	+ 65,000
8	.3333	+ 25,940	−32,479	+ 70,140
9	.3821	− 30,090	−20,015	+ 21,010
10	.4294	+ 11,470	−32,985	+ 68,770
11	.4716	− 90,805	− 4,678	− 24,805
12	.5201	− 95,810	+ 2,756	− 16,810
13	.5668	−137,830	+20,320	− 41,430
Inside Surface	.6132	−184,084	+61,361	− 58,000
Cylinder No. 17 Submerged Flush at 1600° F.				
Outside Surface 1	.0000	− 71,200	+23,730	− 45,500
2	.0475	+ 11,610	− 6,300	+ 37,610
3	.0920	+ 41,330	−10,040	+ 69,930
4	.1429	+ 48,895	−16,210	+ 89,595
5	.1912	+ 10,000	− 7,002	+ 42,900
6	.2472	− 20,200	+ 1,520	+ 16,081
7	.2950	+ 18,312	− 6,150	+ 57,822
8	.3420	+ 36,405	−18,310	+ 80,204
9	.3943	+ 50,053	−22,250	+101,103
10	.4313	+ 78,251	−35,310	+135,325
11	.4823	+ 38,125	−17,241	+104,054
12	.5439	+ 80,850	−25,180	+159,129
13	.5756	+115,206	−40,321	+211,463
Inside Surface	.6154	+150,180	−50,060	+275,180

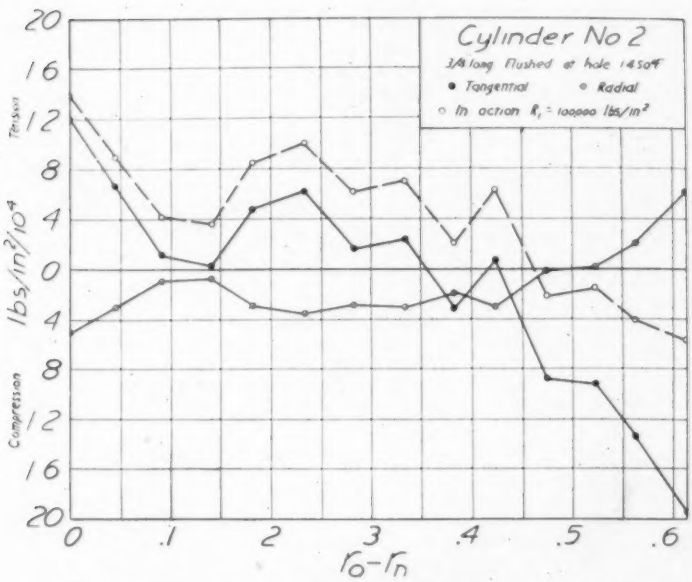


Fig. 8—Chart Showing Stress Distribution Plotted with Wall Thickness as Abscissas. Left End is Outside Surface, Right End is Inside Surface at the Hole. Cylinder ¾ Inch Long Quenched by Flushing at the Hole at 1450 Degrees Fahr.

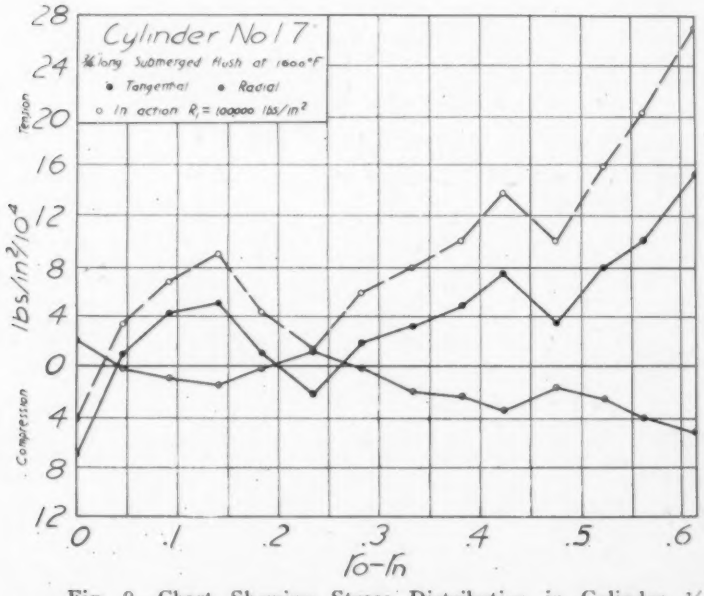


Fig. 9—Chart Showing Stress Distribution in Cylinder ¾ Inch Long Quenched by Submerged Flushing at 1600 Degrees Fahr. Left End is Outside Surface, Right End Inside Surface at the Hole.

The Stress Determinations

The distribution of the tangential and radial stresses was determined in a number of cylinders ¾ inches long. It will be shown

Table VIII
Distribution of Tangential Stress in Cylinders ¾" Long

Layer No.	Flushed at Hole at 1500° F. ($r_o - r_n$) Inches	Flushed at Hole at 1500° F. Tangential Stress Lbs. Per Sq. In.	Flushed at Hole at 1550° F. ($r_o - r_n$) Inches	Flushed at Hole at 1550° F. Tangential Stress Lbs. Per Sq. In.
Outside Surface 1	.0000	+113,250	.0000	+123,800
2	.0509	+ 39,950	.0504	+ 57,820
3	.0947	— 16,860	.0979	+ 21,890
4	.1471	+ 37,107	.1451	+ 49,845
5	.1939	+ 78,280	.1930	+ 66,090
6	.2426	+ 40,173	.2402	+ 69,761
7	.2923	+ 33,773	.2913	+ 31,191
8	.3400	+ 29,074	.3380	+ 23,440
9	.3885	+ 640	.3865	— 46,375
10	.4376	— 24,785	.4339	+ 1,235
11	.4781	— 71,249	.4780	— 89,925
12	.5256	— 98,285	.5242	—101,170
13	.5721	—125,300	.5714	—139,410
Inside Surface	.6192	—173,183	.6178	—195,429

later that the axial stresses need not be considered in these short cylinders. The measurements on which the following calculations are based were made at the center of the holes of the specimens. The results obtained by flushing at the hole at 1450 degrees Fahr. are compared to the results obtained by submerged flushing at 1600 degrees Fahr. in Table VII. These data are shown graphically in Figs. 8 and 9. Abscissas are the differences between the original outside radius and the radii after each grinding. To the observed data there have been added the stresses resulting from the radial pressure of 100,000 pounds per square inch in the hole. The vast difference in type of stress obtained by these two methods of quenching is quite obvious. The tangential stress at the hole of the cylinder quenched by submerged flushing is seen to be in tension, while the hole of the cylinder quenched by flushing the hole is in tangential compression. No explanation of the saw-toothed form of the curves can be offered. It is characteristic of all cylinders and apparently not related to metallographic constituents. Table VIII gives the results obtained with ¾ inch cylinders of carbon tool steel flushed at the hole at 1500 and 1550 degrees Fahr., and Table IX ¾ inch cylinders of 30 per cent nickel steel flushed at the hole at 1450 and 1600 degrees Fahr. The data obtained on the latter are plotted in Fig. 10. The 30 per cent nickel steel of course does not harden on quenching and the holes are merely water annealed. Even the tangential stress in these specimens shows the same saw-toothed form.

The stresses at the hole and at the outside surface of cylinders of each length after various treatments quenched both by submerged flushing and flushing the hole, are given in Tables X, XI, XII and

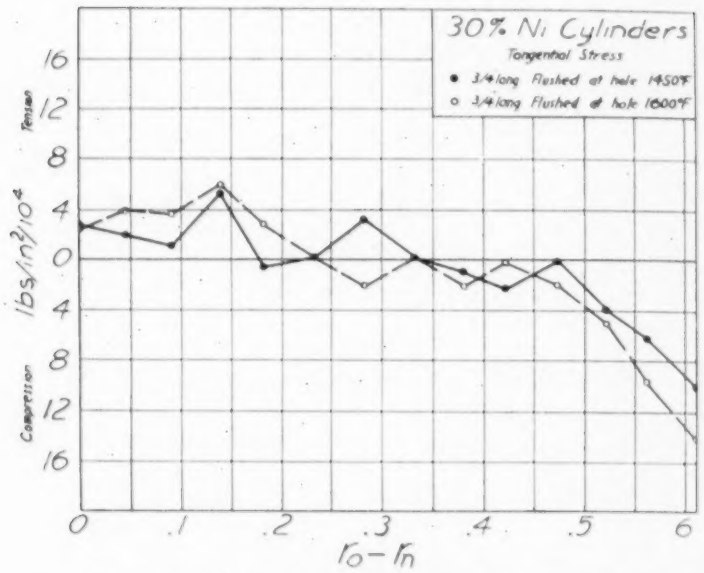


Fig. 10—Chart Showing Distribution of Tangential Stress in 30 Per Cent Nickel Steel Cylinders Quenched by Flushing at the Hole at 1450 Degrees Fahr. and 1600 Degrees Fahr. Left End is Outside Surface, Right End is Inside Surface at the Hole.

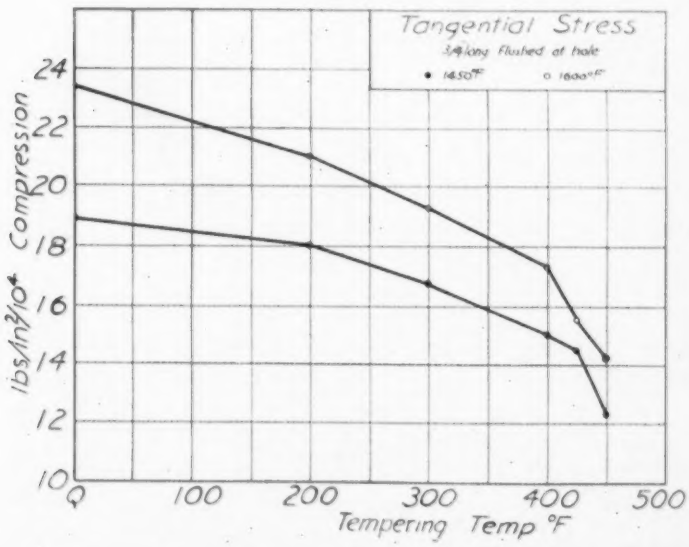


Fig. 11—Chart Showing Decrease in Tangential Stress in Cylinders ¾ Inch Long, Quenched by Flushing at the Hole at 1450 Degrees Fahr. and 1600 Degrees Fahr., Tempered at the Temperatures Indicated.

XIII. The results of tempering cylinders ¾ inches long flushed at the hole are given in Fig. 11. Some of the data presented in Tables X to XIII inclusive are plotted in Fig. 12. These curves show the stress at the center of holes only. The measurements upon which

Table IX

Distribution of Tangential Stress in 30% Nickel Cylinders ¾" Long

Layer No.	Flushed at Hole at 1450° F. $(r_o - r_n)$ Inches	Flushed at Hole at 1450° F. Tangential Stress Lbs. Per Sq. In.	Flushed at Hole at 1600° F. $(r_o - r_n)$ Inches	Flushed at Hole at 1600° F. Tangential Stress Lbs. Per Sq. In.
Outside Surface 1	.0000	+ 31,200	.0000	+ 28,362
2	.0473	+ 19,264	.0472	+ 39,125
3	.0950	+ 14,655	.0934	+ 37,463
4	.1429	+ 55,321	.1431	+ 63,566
5	.1945	− 8,156	.1921	+ 33,174
6	.2425	+ 1,120	.2420	+ 6,805
7	.2951	+ 30,425	.2940	− 24,823
8	.3435	+ 3,196	.3407	+ 2,565
9	.3901	− 10,431	.3896	− 23,215
10	.4380	− 23,563	.4376	− 4,518
11	.4863	− 692	.4845	− 20,330
12	.5354	− 41,253	.5339	− 58,359
13	.5967	− 62,389	.5955	− 95,364
Inside Surface	.6172	−113,000	.6184	−145,217

Table X

Tangential Stress in Cylinders ¾" Long

Treatment: Quenching Temp. Degrees Fahr.	Tempering Temp. Degrees Fahr.	Tangential Stress at Middle of Hole Lbs. Per Sq. In.	Tangential Stress at Outside Surface Lbs. Per Sq. In.
		Flushed at the Hole	
1450		−188,798	+117,523
1500		−196,521	+113,255
1550		−204,364	+123,800
1600		−235,260	+108,436
1450	200	−180,316	+114,525
1450	300	−168,053	+115,213
1450	400	−152,619	+ 92,465
1450	425	−145,538	+ 71,863
1450	450	−123,523	+ 50,808
1600	200	−210,156	+107,342
1600	300	−194,531	+115,268
1600	400	−175,289	+ 86,321
1600	425	−155,614	+ 80,432
1600	450	−143,226	+103,183
		Submerged Flush	
1450		+143,604	−100,516
1600		+150,620	− 71,209
1450	450	+ 59,860	−100,805
1600	300	+ 68,425	− 80,521
1600	450	+ 83,348	− 75,067

these calculations are based are made at point B in Fig. 6. Table XIV gives the values used in Fig. 12.

Discussion of Results

It is interesting to note that cylinders quenched by flushing at the hole show an increase in tangential compression at the hole as the quenching temperature increases. The tangential tension at the hole of cylinders quenched by submerged flushing decreases with increas-

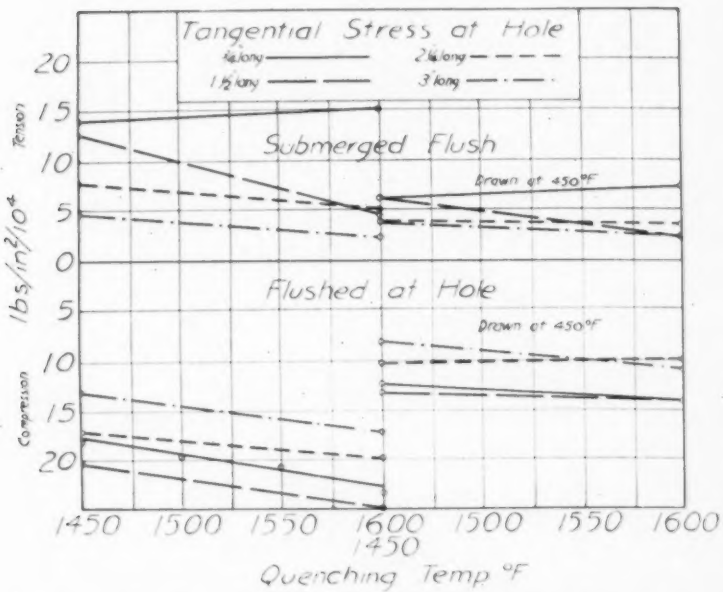


Fig. 12—Chart Showing Influence of Quenching Temperature and Length on Tangential Stress. Cylinders Quenched by Submerged Flushing and Flushing at the Hole. Note Tangential Stresses After Former Treatment, at the Center of the Hole are Tensional, and After the Latter, Compressional.

ing quenching temperature, with the exception of the cylinder ¾ inches long which hardened through at 1600 degrees Fahr. Tempering at 450 degrees Fahr. reduces the magnitude of the stresses in all cylinders irrespective of treatment or temperature. Fig. 11 shows that the decrease in stress on tempering is proportional with both quenching temperatures until 425 degrees Fahr. is reached. From this point on, the curves converge. It would not be unlikely to find the lines of both the 1450 and the 1600 degrees Fahr. treatments meeting at some low stress after tempering at 500 degrees Fahr.

The tangential stress of cylinders quenched by flushing at the hole is seen to be in compression at the top, middle and bottom at both 1450 and 1600 degrees Fahr. The cylinders quenched by submerged flushing at 1450 degrees Fahr., however, showed tangential compression at the ends and tension in the middle. This may be explained by considering briefly the changes taking place during quenching. The ends and outer surfaces of the cylinders quenched by submerged flushing cooled more rapidly than the holes. Consequently the martensitic layer is thicker on the ends and outer surfaces than at the holes. At the point of martensite formation, the layers at the ends and outside surfaces are thick enough so that

Table XI
Tangential Stress in Cylinders 1½" Long

Treatment		Flushed at the Hole		
Quenching Temp. Degrees Fahr.	Tempering Temp. Degrees Fahr.	Tangential Stress at the Hole Lbs. Per Sq. In. Average of Top and Bottom	Middle	Tangential Stress of Outside Surface at Middle of Cylinder Lbs. Per Sq. In.
1450		−230,426	−206,341	+125,361
1600		−243,108	−250,086	+ 90,800
1450	450	−132,485	−133,865	+120,529
1600	450	−160,396	−142,568	+135,488
		Submerged Flush		
1450		−150,945	+130,686	−100,531
1600		+ 43,125	+ 44,840	− 31,342
1450	450	−115,069	+ 60,605	− 80,153
1600	450	− 41,849	+ 19,428	− 30,499

Table XII
Tangential Stress in Cylinders 2¼" Long

Treatment		Flushed at the Hole		
Quenching Temp. Degrees Fahr.	Tempering Temp. Degrees Fahr.	Tangential Stress at the Hole Lbs. Per Sq. In. Average of Top and Bottom	Middle	Tangential Stress of Outside Surface at Middle of Cylinder Lbs. Per Sq. In.
1450		−158,486	−170,531	+113,258
1600		−160,695	−199,680	+120,543
1450	300	−156,095	−145,216	+115,698
1450	450	−126,369	−108,842	+ 96,353
1600	300	−154,645	−165,531	+110,898
1600	450	−122,318	−100,690	+ 85,966
		Submerged Flush		
1450		−147,368	+ 80,880	− 60,594
1600		+110,845	+ 50,608	−120,174
1450	450	− 93,637	+ 40,786	− 85,548
1600	300	− 60,853	+ 56,257	− 43,178
1600	450	− 83,395	+ 42,648	− 53,169

when expansion takes place, the hole is actually warped out of shape. The ends become larger and the middle smaller. The expansion due to the formation of martensite at the center of the hole is naturally much less. Consequently the center of the hole is stretched beyond the normal size and the ends are constrained to a smaller size. Therefore tangential tension is present at the center and tangential compression at the ends. Cylinders quenched by submerged flushing at 1600 degrees Fahr. show uniform tangential tension at the hole. This is probably due to the fact that the hardness penetration at the hole is somewhat deeper than at 1450 degrees Fahr. However, this increase in penetration at the hole is not in proportion to the increase at the ends and outside. Hot upsetting takes place as before, but there is sufficient expansion at the center of the hole to allow the generation of tangential tension in the whole length. In cylinders

quenched by flushing the hole at both 1450 and 1600 degrees Fahr., the formation of martensite at the hole, with its attending expansion, takes place while the unhardened outside is contracting upon it. This naturally throws the martensite at the hole in tangential compression.

It is evident from Fig. 12 that as the length of cylinders increased, the magnitude of the internal tangential stresses decreased.

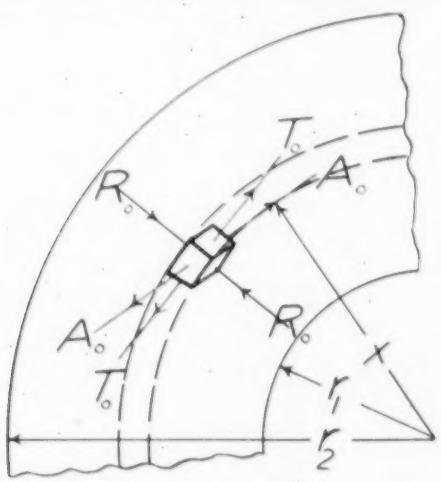


Fig. 13 — Diagram Showing the Stresses at any Point on a Cross Section of the Annulus of a Cylinder.

The only exceptions to this were the cylinders 1½ inches long quenched by flushing at the hole. These cylinders had a slightly greater tangential compression than the cylinders ¾ inch long.

Table XV shows the total length change at the hole on quenching as compared to the total length change at the hole after the removal of the nth layer, i. e., after grinding the cylinder from a wall thickness of 0.625 inch to a thickness of 0.015 inch. The cylinders ¾ inch long show the greatest length change on quenching and no length change on grinding. Consequently there was no axial stress present in these specimens. The longer cylinders show a gradual increase in length change on grinding and decrease in length change on hardening. The flat cylinders expanded to such an extent that there was no axial stress built up. The axial stresses developed in the larger cylinders at the hole are compressional. Evaluation of these stresses shows them to be of little consequence. Thus the axial stresses in cylinders of these proportions may be assumed to be zero. From values given in Table XV, the axial

stresses range from zero to approximately 10,000 pounds per square inch.

The tangential tensional or compressional stresses at the outside of cylinders of this thickness are relatively unimportant. Pressures at the hole result in tangential stresses in the cross-section that are distributed according to a parabolic function. For instance, the pressure of 100,000 pounds per square inch at the hole of a cylinder

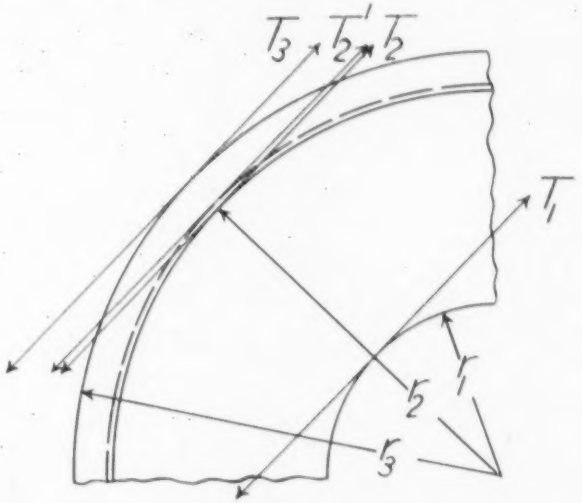


Fig. 14—The Tangential Stresses at Various Points in the Layer Removed and in the Remaining Portion of the Cylinder.

of the dimensions used, results in a tangential tension of approximately 125,000 pounds per square inch at the hole and only 25,700 pounds per square inch tangential tension at the outside surface.

The vast difference in residual stress resulting from flushing at the hole and submerged flushing is quite evident. The stresses in cylinder No. 17, quenched by submerged flushing, are exactly opposite those in cylinder No. 2, quenched by flushing at the hole. The outside of No. 17 is in tangential compression and the inside surface at the hole in tangential tension. In cylinder No. 2 this condition is reversed. The effect of such residual stresses in cylinders at rest can be shown by assuming the cylinder to be in action with a radial force of 100,000 pounds per square inch in the hole. The inside surface of cylinder No. 2 is still in compression even with this force in action in the hole. Consequently the intrinsic strength of the steel at the hole is not called upon to resist this tremendous pressure. Cylinder No. 17 under the same conditions shows a tangential tension at the hole that undoubtedly would cause failure.

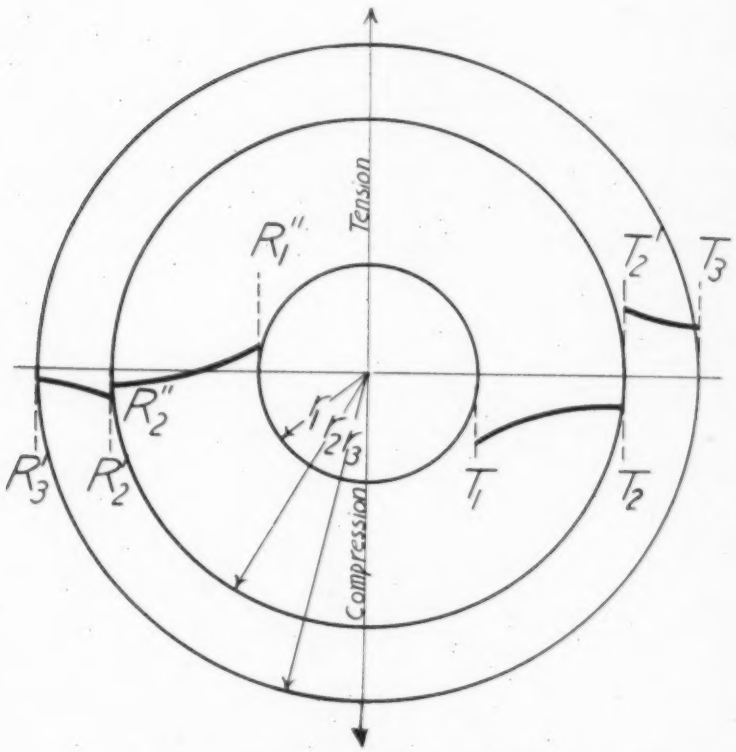


Fig. 15—Stress Distribution After the Removal of the First Layer. Values Plotted from Measurements on a Cylinder ¾ Inch Long, Flushed at the Hole at 1450 Degrees Fahr.

T_1	− 15,100	R''_1	+ 5,030
T_2	− 6,300	R''_2	− 3,760
T'_2	+122,000	R'_2	− 46,400
T_3	+113,000	R'_3	− 37,666
R_2	− 6,620		

Note: Stresses in portion of cylinder remaining after removal of first layer are shown × 10.

The stresses in cylinders quenched by flushing at the hole are, therefore, in the right direction to resist pressures at the hole. Such methods of quenching used for drawing dies or quenching followed by a low temper for header dies, should result in super-tools. The results obtained by L. S. Cope[4] show that the advantages of this treatment are not theoretical but entirely practical. Probably the best known example of using stresses to resist internal pressures is the work of Langenberg.[5] Formerly tangential compression in gun tubes necessary to withstand high powder pressures, were obtained by shrinking a number of hoops on the tube. Langenberg found that by expanding the inner surfaces of gun tubes by cold

[5]F. C. Langenberg, "Effect of Cold Working on the Strength of Hollow Cylinders," TRANSACTIONS, American Society for Steel Treating, Vol. VIII, 1925, p. 447.

Table XIII
Tangential Stress in Cylinders 3" Long

Treatment		Flushed at the Hole		
Quenching Temp. Degrees Fahr.	Tempering Temp. Degrees Fahr.	Tangential Stress at the Hole Lbs. Per Sq. In. Average of Top and Bottom	Middle	Tangential Stress of Outside Surface at Middle of Cylinder Lbs. Per Sq. In.
1450		−143,427	−130,675	+115,468
1600		−160,539	−173,490	+123,887
1450	300	−107,068	−100,694	+ 86,428
1450	450	−104,563	− 80,320	+ 89,659
1600	450	−111,552	−109,360	+ 93,103
		Submerged Flush		
1450		−140,521	+ 48,486	− 63,158
1600		+ 59,368	+ 19,548	− 48,367
1450	450	−130,168	+ 39,683	− 39,985
1600	300	− 60,421	+ 10,546	− 20,495
1600	450	− 86,667	+ 20,484	− 53,364

Table XIV
Comparison of Tangential Stress Obtained by Both Methods of Treatment

	Stresses at Middle of Holes Only As Quenched — Degrees Fahr.			
Length of Cylinder Inches	Flushed at Hole 1450	Submerged Flush 1450	Flushed at Hole 1600	Submerged Flush 1600
¾	−188,798	+143,604	−235,260	+150,620
1½	−206,341	+130,686	−250,086	+ 44,840
2¼	−170,531	+ 80,880	−199,680	+ 50,608
3	−130,675	+ 48,486	−173,490	+ 19,543
		Drawn at 450° Fahr.		
¾	−123,523	+ 59,860	−143,226	+ 83,348
1½	−133,865	+ 60,605	−142,568	+ 19,428
2¼	−108,842	+ 40,786	−100,690	+ 42,648
3	− 80,320	+ 39,663	−109,360	+ 20,484

Table XV
Length Change at the Hole

	Quenched at 1450° F.			
	Submerged Flush		Flushed at Hole	
Length of Cylinder Inches	As Quenched Based on Original Length Inches	After Removal of nth Layer Based on Length as Quenched Inches	As Quenched Based on Original Length Inches	After Removal of nth Layer Based on Length as Quenched Inches
¾	+.0033	.0000	+.0031	.0000
1½	+.0012	+.0005	+.0020	+.0001
2¼	+.0013	+.0007	+.0005	+.0005
3	+.0003	+.0010	+.0002	+.0011
	Quenched at 1600° F.			
¾	+.0042	.0000	+.0033	+.0001
1½	+.0028	+.0005	+.0030	.0000
2¼	+.0025	+.0007	+.0025	+.0006
3	+.0020	+.0008	+.0021	+.0012

working, the stresses so formed induced tangential compression in the inner surfaces. Consequently the necessity for shrinking hoops

on gun tubes was eliminated. Incidentally the form of the curves showing the distribution of the tangential stress in the section of gun tubes so treated are the same as those of a cylinder quenched by flushing at the hole.

Since the stresses in dies quenched at the hole have been employed to advantage, and the metal back of the hardened zone is soft and tough, there is no reason to reduce the stresses by tempering for certain types of applications. For header dies, however, it would probably be necessary to temper such a die in order to reduce the possibility of cracking or spalling and allow reaming. However, as shown in Fig. 11, the residual tangential compressional stress in a die quenched at the hole even after tempering at 450 degrees Fahr. is of considerable magnitude. Obviously it has been found absolutely necessary to temper dies quenched by submerged flushing in order to reduce the tangential tension to a minimum.

Summary

A summary of some of the more important results of this work is as follows:

1. The axial compression in the outside surface of quenched solid bars decreases with increase of hardness penetration, but increases with quenching temperature except in cases of complete hardening of the section.

2. The portions of cylinders under martensitic layers formed by quenching at 1600 degrees Fahr. are considerably harder than those under layers formed by quenching at 1450 degrees Fahr.

3. The tangential compression at the holes of cylinders quenched by flushing at the hole increases with quenching temperature. The tangential tension in the center of the holes of cylinders quenched by submerged flushing decreases with increase of quenching temperature.

4. The tangential compression at the holes of cylinders quenched by flushing at the hole decreases with increase of the length of the cylinders. The tangential tension at the center of holes of cylinders quenched by submerged flushing also decreases with increase of the length of the cylinders.

5. The holes of cylinders quenched by flushing at the hole are uniformly in tangential compression even after tempering at 450 degrees Fahr. The holes of cylinders quenched by submerged flush-

ing at 1450 degrees Fahr. are in tangential tension at the center and in tangential compression at the ends. This condition persists after tempering at 450 degrees Fahr. The holes of cylinders quenched by submerged flushing at 1600 degrees Fahr. are uniformly in tangential tension. Tempering at 450 degrees Fahr. after submerged flushing at 1600 degrees Fahr. leaves the center of the holes in tension, but changes the stress at the ends to tangential compression.

6. The axial stresses when present at the holes of cylinders are compressional. This stress increases with increase of length of cylinders, but never reaches values that are of consequence.

7. A tabulation of the differences between the two methods of quenching employed is as follows:

Flushed at the Hole	Submerged Flushing
Little or no warping.	Considerable warping.
Uniform tangential compression at the hole.	In some cases both tangential compression and tension are present in the hole; in others uniform tangential tension in the hole.
Stress in right direction to resist pressure at the hole.	Stresses at center of hole in tension and therefore in wrong direction to resist pressures at the hole.
May be used in untempered condition for some purposes; however, tempering as high as 450 degrees Fahr. leaves considerable tangential compression at the hole.	Must be tempered to relieve tensional stresses. Compressional stresses at the ends are also reduced. In all cases after tempering tangential tension remains in the centers of the holes.
Intrinsic strength of steel at the hole not used to resist pressures in the hole.	Actual strength of the steel at the hole must be used to resist pressures in the hole.
The extreme hardness of quenched martensite may be retained in the hole for certain applications such as drawing dies.	Holes and backing are considerably softer due to the necessity of tempering.

The information reported here represents the investigation of one particular problem. Each different type of tool is an individual case. However, an application of the basic principles employed in this work should yield considerable information in regard to the direction and magnitude of the residual quenching stresses. If it is found by such analysis that the quenching stresses have not been used to advantage, new quenching methods may be derived. If the

quenching is adjusted so that the resulting stresses are beneficial, fewer tools will be lost during heat treatment, and substantial increases in performance will be realized. If this paper does nothing more than to stimulate interest in the field of scientific quenching, its purpose will have been achieved.

The author wishes to acknowledge his indebtedness to G. V. Luerssen and F. R. Palmer for much helpful criticism and comment, and to express his appreciation of help given him by members of the metallurgical department.

APPENDIX I
HEYN'S METHOD FOR THE ESTIMATION OF AXIAL STRESS

A brief summary of Heyn's method for the determination of axial internal stresses is as follows:

The length change of a bar with a circular cross section is measured as thin layers are successively removed from the surface. If it is assumed that the length of the specimens increases on the removal of the first layer, this layer must have been in tension and the remainder of the bar in compression. The forces causing the bar to be elastically decreased in length in its original condition have now been eliminated. The axial stress in the first layer is therefore,

$$S_1 = E \frac{f''_1}{f'_1} \frac{L_1 - L}{L}$$

Where,

f'_1 = the cross section of the first layer
f''_1 = the cross section of remaining portion
L = original length
L_1 = length after removal of first layer
S_1 is tensional when L_1 is greater than L, and compressional when L_1 is less than L.

The calculation of the stress in the second layer and succeeding layers must take the effect of preceding layers into consideration. The general equation is:

$$S_n = \frac{E}{L} \cdot \frac{f''_n (L_n - L) - f''_{n-1} (L_{n-1} - L)}{f'_n}$$

The residual stress after the nth layer has been removed:

$$S = \frac{E}{L} (L - L_n)$$

E — Modulus of elasticity
f'_n — cross section of nth layer
f''_n — cross section remaining after removal of nth layer
L — original length
L_n — length of specimens with cross section f''_n

For a complete discussion of this method see the original source.[3]

APPENDIX II

METHOD FOR THE ESTIMATION OF THE DISTRIBUTION OF INTERNAL TANGENTIAL AND RADIAL STRESSES IN HOLLOW CYLINDERS

As previously mentioned, thin layers were successively removed from the circumference of the cylinders and the changes in inside diameters measured. The derivation of formulas by which these observations may be used to determine the distribution of the tangential and radial stresses is as follows. Much of this theory is an application of basic principles as given by Merriman.[6] His nomenclature has been generally followed. The author is therefore deeply indebted to this work.

Consider a thick hollow cylinder subject to a pressure R_1 on each square unit of inside surface and R_2 on each square unit of outside surface r_1 and r_2 —inside and outside radii respectively.

The length of the cylinder must be considered as somewhat larger than the outer diameter so that the disturbing influence of the ends may not affect the reasoning.

Let x be the distance from the axis of the cylinder to any point in the x-section of the annulus, not near the end, Fig. 13. Any elementary particle is held here in equilibrium by the longitudinal unit stress A_o, a tangential unit stress T_o, and a radial unit stress R_o. The value of R_o is taken as being somewhere between R_2 and R_1. However, both R_o and T_o may be regarded as tensile. R_o shown in Fig. 13 is in compression. From the theories of true stress the effective longitudinal unit elongation ϵ of the cylinder due to these stresses is:

$$\epsilon = \frac{(A - \Phi T_o - \Phi R_o)}{E}$$

Φ = factor of lateral contraction (Poisson's Ratio, 1/3 for steel)
E = modulus of elasticity

Therefore:

$$\epsilon_{aE} - \text{True axial} = A_o - \frac{T_o}{3} - \frac{R_o}{3}$$

$$\epsilon_{rE} - \text{True radial} = R_o - \frac{T_o}{3} - \frac{A_o}{3}$$

$$\epsilon_{tE} - \text{True tangential} = T_o - \frac{R_o}{3} - \frac{A_o}{3}$$

Both A and ϵ are constant for all parts of the annulus, since the cylinder has been assumed as larger than its diameter. This condition is true at some point not near the ends. Consequently it follows that T_o and R_o = Constant, or

$$T_o + R_o = 2\,C_1 \qquad (1)$$

It shall be noted that $T_o + R_o$ = constant, when A is constant as well as when A = O.

An elementary annulus of thickness dx is shown. The inner radius is x, and its outer is x + dx. The pressure normal to any diameter at the

[6] M. Merriman, "Mechanics of Materials," John Wiley & Sons, 1916.

inner surface is $R_o x$ — (for pipes under internal pressure) ts = rR, and $(R_o + dR_o)(x + dx)$ for the outer surface of the elementary annulus. Equation for equilibrium where acting pressure equals resisting stress is:
$(R_o + dR_o)(x + dx) - R_o x = T_o\, dx$ (as above ts = rR), i. e., destructive forces and resistance are equal,
or
$x dR_o + R_o dx = T_o dx$ (dx dR_o is considered so small as to be negligible)

$$x dR_o + R_o dx = (2 C_1 - R_o)\, dx$$

$$\frac{x dR_o}{dx} + R_o = (2 C_1 - R_o)$$

$$\frac{x dR_o}{dx} = 2 C_1 - 2 R_o$$

$$\frac{X}{2\, dx} = \frac{C_1 - R_o}{dR_o} \quad \text{or} \quad 2 \int \frac{dx}{x} = \int \frac{dR_o}{C_1 - R_o}$$

Integrating:

$$R_o = C_1 - \frac{C_2}{x^2} \qquad (2)$$

Stress distribution is parabolic. Lamé's formula

Placing value of R_o in (1)

$$T_o = C_1 + \frac{C_2}{x^2}$$

The constant of integration C_2 is to be evaluated by assuming the limiting values for R, i. e., R_1 and R_2; $R_o = -R_1$ when $x = r_1$; $R_o = -R_2$ when $x = r_2$. Inserting these values in (2)

$$C_1 = \frac{r_1^2 R_1 - r_2^2 R_2}{r_2^2 - r_1^2}$$

$$C_2 = \frac{r_1^2 r_2^2 (R_1 - R_2)}{r_2^2 - r_1^2}$$

Inserting these constants in (2):

$$R_o = \frac{r_1^2 R_1 - r_2^2 R_2 - \frac{r_1^2 r_2^2}{x^2}(R_1 - R_2)}{r_2^2 - r_1^2} \qquad (3)$$

$$T = \frac{r_1^2 R_1 - r_2^2 R_2 + \frac{r_1^2 r_2^2}{x^2}(R_1 - R_2)}{r_2^2 - r_1^2} \qquad (5)$$

(3) and (4) are Lamé's formulas for tangential and radial unit stresses under inside and outside pressures. These formulas refer to apparent stresses only and have been deduced for a hollow cylinder with closed ends. With the cylinders used there are no closed ends or inside pressures, hence there is no external longitudinal pressures on them. Accordingly the axial unit stress is zero. The true tangential unit stress $T = T_o - \frac{1}{3} R_o$. Using the values for T_o and R_o given above in (3) and (4):

$$T_o = \frac{2 r_1^2 R_1 - 2 r_2^2 R_2 + \frac{4 r_1^2 r_2^2}{x^2}(R_1 - R_2)}{3 (r_2^2 - r_1^2)} \qquad (4)$$

which is Birnie's formula.

In the present problem the thin layers removed can be regarded as hoops that are eliminated from the system as grinding progresses. Incidentally all grinding was done wet. The expansions or contractions of r_1 are measured and corrected to 20 degrees Cent. In order to calculate the stresses causing this radius change, the formulas previously developed may be used. Suppose a compound cylinder to be formed by shrinking a hoop on a tube. As before the inner radius of the tube is r_1, the outside radius of the tube and the inside radius of the hoop r_2, and the outside radius of the hoop r_3. In consequence of the shrinkage, a radial unit pressure R_2 is produced between the two surfaces. This causes the tube to be in tangential compression and the hoop to be in tangential tension.

The following nomenclature shown in Fig. 14 is used:

T_1 — tangential compression at bore of tube
T_2 — tangential compression at outer surface of the tube
T'_2 — tangential tension at inner surface of hoop
T_3 — tangential tension at outer surface of hoop
R_2 — radial pressure between layers

If e is the length change of any radius x, $2\pi e$ is the circumferential length change of any circumference $2\pi x$. $\frac{2\pi e}{2\pi x}$ is the unit length change due to the unit stress T. Thus $\frac{e}{x} = \frac{T}{E}$. When $x = r_1$ and e is the length change of the inner radius of the tube,

$$-T_1 = \frac{e\,E}{r_1} \qquad (6)$$

for the tangential unit stress (compression in this case) at the bore of the tube. The tangential compression at the outside of the tube in (5) let

$$R_1 = 0 \text{ and } x = r_2,$$

then

$$-T_2 = R_2 \cdot \frac{2}{3} \cdot \frac{2\,r_1^2 + r_2^2}{r_2^2 - r_1^2}$$

Let

$$\frac{2}{3} \cdot \frac{2\,r_1^2 + r_2^2}{r_2^2 - r_1^2} = \alpha \text{ then } T_2 = \alpha\, R_2 \qquad (7)$$

The tangential tension at the inside of the hoop T'_2 may also be found from (5) by replacing R_1 by R_2, R_2 by O, r_1 by r_2 and r_2 and r_3, then:

$$T'_2 = R_2 \cdot \frac{2}{3} \cdot \frac{r_2^2 + 2\,r_3^2}{r_3^2 - r_2^2}$$

Let

$$\frac{2}{3} \cdot \frac{r_2^2 + 2\,r_3^2}{r_3^2 - r_2^2} = \beta \text{ then } T'_2 = \beta\, R_2 \qquad (8)$$

Dividing (7) by (8)

$$T'_2 = \frac{\beta}{\alpha} T_2 \qquad (9)$$

However, T_1 in terms of T_2 may also be found from (5) by substituting the value of R_2 now known (7) and making $x = r_1$, which results

$$T_2 = T_1 \frac{2\ r_1^2 + r_2^2}{3\ r_2^2} \qquad (10)$$

(This equation determines the distribution of the stress in the tube. Inspection shows it to be parabolic).

In a like manner T_3 can be found from (5) where $R_1 = R_2$; $R_2 = R_3$; $r_1 = r_2$; $r_2 = r_3$.

$R_3 = 0$ and $x = r_3$, then

$$T_3 = \frac{2\ R_2\ r_2^2}{r_3^2 - r_2^2} \qquad (11)$$

Recapitulating:

$$T_1 = \frac{e\ E}{r_1} \quad \text{Tangential stress — inner surface of tube}$$

$$T_1 = T_2 \frac{3\ r_2^2}{(2\ r_1^2 + r_2^2)} \quad \text{Solve for } T_2 \text{ — Tangential stress — outer surface of tube}$$

$$T'_2 = \frac{\beta}{\alpha} T_2 \quad \text{Tangential stress — inner surface of hoop}$$

$$R_2 = \frac{T_2}{\alpha} \text{ or } \frac{T'_2}{\beta} \quad \text{Radial pressure between layers}$$

$$T_3 = \frac{2\ R_2\ r_2^2}{r_3^2 - r_2^2} \quad \text{Tangential stress — outer surface of hoop}$$

$$\alpha = \frac{2}{3} \frac{2\ r_1^2 + r_2^2}{r_2^2 - r_1^2}$$

$$\beta = \frac{2}{3} \frac{r_2^2 + 2\ r_3^2}{r_3^2 - r_2^2}$$

All values necessary are known from the observed value of T_1.

As the number of layers increase, it is necessary to imagine each successive layer to have been acted upon by the previous layers. For instance, layer 4 was acted upon by layers 1, 2 and 3. If layers 1, 2 and 3 were in tangential tension, then the observed tangential tension in layer 4 must have been diminished by the tangential compressions caused by layers 1, 2 and 3 in the original specimen. Since the stress distribution is parabolic, equation (10) may be used to determine the tangential compression in any layer. The sum of the tangential compressions caused by each preceding layer is then subtracted from the tangential tension in the outside of the layer under observation and the result represents the actual stress existing at the outside of that layer in the original specimen.

For example, the tangential tension at the outside of the fourth layer of a cylinder was found to be 20,650 pounds per square inch. However, in the original cylinder, layer 1 imposed a tangential compression of 7900 pounds per square inch in layer 4, layer 2 imposed a tangential compression of 5420 pounds per square inch, and layer 3, of 1600 pounds per square inch. The sum of these is 14,920 pounds per square inch tangential compression in layer 4, caused by layers 1, 2 and 3. The difference between this and 20,650 pounds per square inch is 5,730 pounds per square inch tangential tension at the outside of layer 4 in the original specimen.

Calculation of Radial Stresses

The true radial stresses may also be evaluated from equations 3 and 4. As before, since the axial stress is zero, the true radial stress $R = R_o - \frac{1}{3} T_o$. Substituting the values of R_o and T_o given in (3) and (4) and making $R_1 = O$, there results the following general equation:

$$R = \frac{2 R_2 \frac{(2 r_1^2 r_2^2)}{x^2} - r_2^2}{3 (r_2^2 - r_1^2)} \qquad (12)$$

Equation (12) may be used to determine the stress distribution in the inner portion or tube. For specific instances such as the outer and inner surfaces of the tube (12) becomes:

Outer surface $x = r_2$

$$R''_2 = \frac{2 R_2 (2 r_1^2 - r_2^2)}{3 (r_2^2 - r_1^2)} \qquad (13)$$

Inner surface $x = r_1$

$$R''_1 = \frac{2 R_2 r_2^2}{3 (r_2^2 - r_1^2)} \qquad (14)$$

The same equations are applied to the hoop. If all the transcripts of (3) and (4) are increased by unity, and $R = R_o - \frac{1}{3} T_o$ as before, the following results with $R_2 = O$:

General Equation:

$$R = \frac{R_2 \left(2 r_2^2 - 4 \frac{(r_2^2 - r_2^2)}{x^2} \right)}{3 (r_2^2 - r_2^2)} \qquad (15)$$

However, R''_1 may be found in terms of R''_2 since both are values of R_2. Substituting the value of R_2 from (13) in (14) the following results:

$$R''_1 = R''_2 \cdot \frac{r_2^2}{(2 r_1^2 - r_2^2)} \qquad (16)$$

This equation also determines the distribution of stress in the tube.

Specific cases as for outer and inner surfaces of the hoop (15) becomes:

Outer surface $x = r_3$

$$R'_2 = - \frac{2 R_2 r_2^2}{3 (r_3^2 - r_2^2)} \qquad (17)$$

Inner surface $x = r_2$

$$R'_2 = \frac{2 R_2 (r_2^2 - 2 r_3^2)}{3 (r_3^2 - r_2^2)} \qquad (18)$$

RECAPITULATION:

R_2 = Radial pressure between contact surfaces.

$$R''_1 = \frac{2\ r_2^2\ R_2}{3\ (r_2^2 - r_1^2)}$$ Radial stress — Inner surface tube.

$$R''_2 = \frac{2\ R_2\ (2\ r_1^2 - r_2^2)}{3\ (r_2^2 - r_1^2)}$$ Radial stress — Outer surface tube

$$R'_2 = \frac{2\ R_2\ (r_2^2 - 2\ r_3^2)}{3\ (r_3^2 - r_2^2)}$$ Radial stress — Inner surface hoop

$$R'_3 = -\frac{2\ R_2\ r_2^2}{3\ (r_3^2 - r_2^2)}$$ Radial stress — Outer surface hoop

$$R''_1 = R''_2\ \frac{r_2^2}{2\ (r_1^2 - r_2^2)}$$ Radial stress — Stress distribution in tube.

The distribution of internal radial stresses across the wall of a cylinder in its original state may be found by a method similar to that employed for tangential stress. As the layers increase, it is necessary to take into consideration the effect of each preceding layer. The pressures between the layers have been designated by R_2. Using the value of R_2 found between any layer and the remaining portion of the cylinder, the radial stress at any point in this remaining portion may be found by equations 13, 14 and 16. Thus the influence of preceding layers may be evaluated. For example the radial stress at the outside of the seventh layer of a cylinder was found by equation 17 to be −24569 pounds per square inch (R_2 used was between seventh layer and remaining portion of cylinder). The pressure between layers 1 and 2 cause a radial stress at this point of −2640 pounds per square inch; layers 2 and 3, −1310 pounds per square inch; layers 3 and 4, −1365 pounds per square inch; layers 4 and 5, −782 pounds per square inch; layers 5 and 6, −4020 pounds per square inch; and layers 6 and 7, −2780 pounds per square inch. The sum of these values is −12897 pounds per square inch. Therefore the internal radial pressure in the original cylinder at a point within the wall represented by the outside of layer No. 7 is −37,466 pounds per square inch.

The distribution of the tangential and radial stresses in a cylinder after the removal of the first layer is shown in Fig. 15. The thickness of this layer is of course greatly exaggerated. Actual values of the stresses are given.

If the distribution of stresses across the wall of a cylinder is not of particular interest, and only the magnitude and direction of the forces at the hole of the cylinder is desired, a very simple method may be used to estimate the stress conditions. The diameter of the hole of the cylinder is measured after quenching. The cylinder is then ground down to the thinnest wall possible to obtain, and the diameter change of the hole determined. For example, in the work reported here, cylinders with a 5/8 inch wall were ground to a wall thickness of less than 1/64 inch. The total increase or decrease in the diameter of the hole after grinding represents the release of stress which caused the deformation at this point in the original cylinder. The tangential stress eliminated may be evaluated by the following formula:

$$T = \frac{d - d_1}{d}\ E \qquad (19)$$

d — original diameter of the hole
d_1 — diameter after grinding
E — modulus of elasticity

If d_1 is greater than d, the hole was in tangential compression in the original cylinder, and if d_1 is less than d, the hole was in tangential tension.

DISCUSSION

Written Discussion: By Howard Scott, research department, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

Probably no subject concerned with the hardening of steel is more actively discussed and at the same time less investigated than is that of internal stresses. Those stresses are known to exist in practically all hardened tool steel and are probably present to some degree in most heat treated structural steel. Lacking simple and effective means for their evaluation, it is not surprising that responsibility for failures is often assigned rightly or wrongly to them. In view of the restriction they offer to the full use of the inherent properties of steel, their contribution to failure and the value of property jeopardized an extensive research program in this field is fully justified. Such a program, however, will hardly be undertaken until the serious economical aspects of the matter and the possibility of corrective measures are demonstrated by papers such as Mr. Greene's.

Before discussing the material presented, it is desirable to point out that internal stresses as a result of quenching are caused by two rather distinct phenomena. When a homogeneous material having no transformation in the temperature range involved, such as Mr. Greene's 30 per cent nickel steel, is quenched internal stresss are developed by temperature gradients acting in conjunction with the thermal expansion and plasticity of the metal. Internal stresses developed in that manner might be more properly identified as residual stresses because they are the outcome of stress variations occurring at high temperatures.

The other type of internal stress is caused by non-homogeneity of structure. Its genesis may be well illustrated by steel which is austenitic at atmospheric temperature, but starts to transform into martensite at a slightly lower temperature. If a bulky piece of such steel is plunged in liquid air for a short time and brought back to atmospheric temperature quickly, a surface layer of martensite will have formed. As the specific volume of the martensite is much greater than that of the austenite, the surface will be under compressional stress of high order. In this case no stresses of the type first described will be developed because of the high elastic strength of the metal at low temperature.

Now the point that I have in mind is probably already clear. It is that the internal stresses in superficially hardened steel, such as the carbon tool with which Mr. Greene is concerned, are a resultant of the two types of generating factors just distinguished. The martensitic surface layer on quenched carbon tool steel has a considerably greater specific volume than the pearlitic core though the difference is not so great as that between martensite and austenite in the illustration cited. Consequently, it produces internal stresses of the same sign as in a water-quenched specimen of simple shape having a homogeneous structure. It is not possible to go much farther with the analysis of internal stresses in superficially hardened steels because of the very complex manner

in which they are generated except to point out that the volumetric factor is probably the dominant one controlling the magnitude and distribution of internal stress in this case. This conclusion is based on the observation that the magnitude of the stresses in superficially hardened steel is much greater than in fully hardened steel, both water-quenched and otherwise comparable. I believe, therefore, that one is justified in accepting that view as a guide for the investigation of internal stresses in superficially hardened steel.

Besides giving the answer to a practical hardening problem, Mr. Greene has provided some valuable fundamental information. I refer to his experiments with 30 per cent nickel steel and his determination of internal stress release on tempering. The first mentioned experiments provide information which aids in the relative evaluation of the several factors contributing to internal stress development. The last mentioned experiments are notable because they provide for the first time quantitative information on the reduction of internal stresses in superficially hardened steel. It might be assumed that his data on tempering apply also to fully hardened steel, but the validity of that assumption is questionable in view of the distinctions already made between these two cases.

In regard to the evaluation of internal stresses, Mr. Greene might be a little more explicit. This is a difficult and exacting task requiring dimensional measurements with an accuracy close to plus or minus one hundred thousandth of an inch. Changes of much greater magnitude than this occur with time in steel quenched but not tempered. The dimensional measurements required for internal stress evaluation extend necessarily over a considerable period of time so error due to dimensional changes with time is quite probable unless precautions are taken to avoid it. Whether or not Mr. Greene has achieved the standard of accuracy I indicate, he has performed a meritorious service, the extensive labor behind which is masked by his terse and easily assimilable presentation of results.

Written Discussion: By H. B. Pulsifer, metallurgist, Ferry Cap and Set Screw Co., Cleveland, Ohio.

Any fundamental investigation of die steel like that presented by Mr. Greene should be welcomed by those engaged in the cold heading industry and of use to both them and die steel makers. It is doubtful if the results can be of immediate benefit but it is a good start in the right direction and should be continued to the advantage of all concerned.

It is, of course, reassuring to learn that the stresses in a quenched hollow cylinder are in the right direction to resist pressures in the hole. That determination checks with the fact that the tendency is for the holes to contract on treatment. Their walls would necessarily be under compression.

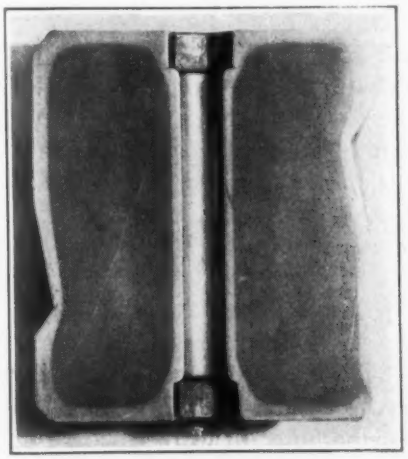
But other details of the investigation are somewhat removed from the conditions of actual practice. To repeat an illustration from this author's discussion of Mr. Jameson's paper we see that a typical die from plant procedure is hardened and has a thick case about the entire exposed surface.

This may not conform to the practice in all plants but we have found it the best practice after exhaustive trials with many sorts of quenching and die construction.

Stresses of considerably different direction and intensity may be found in these dies when compared with what Mr. Greene finds in his cylinders.

Mr. Jameson was mystified by the ability of steel manufacturers to control the depth of hardening while Mr. Greene appears to have that under control, but does not disclose more details. Doubtless more facts will come to light with continued investigation.

Mr. Greene's work is to be greatly commended for this sort of investigation is laborious, requiring much practice and skill. We hope it will be continued for it cannot but be beneficial for the cold headers. It will also reflect to the advantage of the steel makers inasmuch as the cold heading industry appears



Section Through a Split Die 3.5 Inches Long Etched with Picric and Nitric Acids.

to be in direct line with one of the main trends in the metal working activities of the world. The economics of cold heading requires substantial die life, as Mr. Jameson stated, and the success of the industry appears largely in the hands of the steel manufacturers.

Written Discussion: By H. G. Keshian, metallurgist, Chase Companies, Bridgeport, Conn.

To any one who is endeavoring to put the so-called art of heat treating on the sound basis of science, Mr. Greene's paper is of much interest and practical value. It is indeed another step further towards that end where ultimately it is hoped the laws of physics and chemistry will be the guiding spirit of all operations concerned with the treatment of metals; and the more papers of this nature the nearer we would be to that end.

What these quenching stresses are capable of doing is well known to many of us who at times have been surprised to find a tool in many pieces on the bench after it had been hardened and the "stresses relieved" in the best way the experienced heat treater knows how; yet I would like to mention here another but rather a costly example of the way these elusive stresses behave, showing how important it is to press the scientific study of their behavior.

Recently, a company of wide experience in the manufacture of forgings and

modern methods of heat treatment made some hollow cylinders from alloy steel forgings. These measured 33 inches in diameter, 35 inches in length with bores varying from 6½ to 8 inches in diameter. All were heat treated in oil by submerged flushing at the hole. Then they were tempered at a temperature well above that which to all calculations appeared to be sufficient to relieve unbalanced stresses. Almost all of the cylinders cracked open either in transit or soon after they were put into service where they were subjected to an internal pressure of 80,000 pounds per square inch. The cylinders themselves, as indicated by hardness tests made on the split section, showed tensile strength of 120,000 pounds per square inch near the bore and 160,000 pounds near the outside edge with a corresponding yield point of 100,000 and 140,000 pounds. One can imagine the immensity of these quenching stresses that must have been set up in these cylinders. They were so great that they were sufficient to snap open a section of metal 13½ inches thick and 35 inches long having the above physical properties.

Mr. Greene's experiments showed that the flushing the cylinders at the hole will result in the formation of tangential compression and is a safer and better method of quenching header and drawing dies and is preferable to submerged flushing which causes tangential tension which may result in both quenching and service failures. This is in agreement with my experience with header and drawing dies. Submerged flushing, as shown in Fig. 7, in Mr. Greene's paper, causes more distortion of the hole and in shops where this method of quenching is practiced it is almost always necessary to ream the hole. This means that the die has to be tempered considerably to allow reaming with attendant danger to soften the die. If on the other hand the die has been quenched by flushing at the hole the work of reaming is reduced to a minimum, and the dies, in many cases can be used without reaming. There are now, simple, portable devices on the market for this type of quenching.

Mr. Greene, explaining the formation of tangential stresses, mentions the effect of martensite as being responsible for the development of these stresses. This, of course, is the view generally held in explaining the development of quenching stresses, which, if expressed in a different way, would mean that if the steel had no phase change there would not be volume change upon quenching, and if no volume change took place there would be no quenching stresses. If I am reasoning this relation between quenching stresses and phase changes correctly, then the formation of martensitic structure is to be looked for as the source of trouble; but it would seem that neither the formation of martensitic structure nor the phase change is essential for volume change, therefore, for quenching stresses. In the first place, from Fig. 10, in Mr. Greene's paper, it is evident that 30 per cent nickel steel which does not harden upon quenching developed quenching stresses. In the second place, there are metals which do not have phase change, exhibit volume change upon heating and stresses upon quenching. Among these might be mentioned brass and copper and other nonferrous metals some of which even develop cracks in quenching.

It would appear that we would have the problem of quenching stresses whether the steel had phase change or not although perhaps not to the same extent that we have now where the formation of martensitic structure plays an

important part. If this is so and the volume change must depend on other causes, such as atomic movement under the effect of heat, besides the phase change, then the study of quenching stresses or the failures resulting from them, it seems to me, can be carried on to better advantage, if the phase change or the part played by martensite, although very important, was not considered the sole cause of quenching stresses.

Author's Closure

The author wishes to thank Mr. Pulsifer for his discussion. While his results do not entirely agree with the theory developed in the paper, it is quite possible that they can be accounted for. The sections which were used were ideal, and contained straight holes with no off-sets, as header dies usually do. Furthermore the influence of hardness penetration is probably considerably greater than is generally supposed. For instance, in Table II, the deep hardening steel generated much less axial compression in the outer layers than the shallower hardening steels. If the work on cylinders were repeated with shallower or deeper hardening steels, the results very probably would be quite different. It is interesting to learn that Mr. Keshian's experience with header and drawing dies agrees with the contentions of the author in regard to the method of heat treatment.

Mr. Keshian further brings out a rather important point in regard to the generation of stress. The generation of stress is not due to the formation of martensite alone. The 30-per cent nickel steel cylinders shown in Fig. 10 were included to show that martensite was not entirely the cause of stress formation, and that it was partially due to the temperature gradients acting in conjunction with the thermal expansion and hot upsetting. Of course, one school of thought connects the formation of martensite with the generation of stress. That is, without the formation of stress during cooling, the ability to form martensite would be impossible. In Mr. Scott's very scholarly discussion the answer to Mr. Keshian's question is brought out much more clearly.

The author is indebted to Mr. Howard Scott for his very valuable contribution in the discussion of this paper. Measurements were made as Mr. Scott outlines. The change in diameter of the holes were read to plus or minus one hundred thousandth of an inch. Specimens were ground wet so that no heat was generated during the process. After grinding, specimens were placed in a constant temperature chamber, and allowed to stand for 48 hours before measurements were taken. When the temperature in this chamber varied, corrections were made. All results were calculated at 20 degrees Cent. After quenching, specimens were allowed to remain at room temperature for 4 days before measurements were taken. This was decided upon after experimenting with size change on deep, medium and shallow hardening steels. After standing 2 days, the lengths became fairly constant; after 4 days lengths were entirely constant.

EFFECTS OF PREQUENCH TREATMENTS ON THE HARDNESS, BODY, AND STRUCTURE OF HARDENED TOOL STEEL

By R. H. Harrington

Abstract

Difficulties in the forms of low hardness and cracks are often encountered in the heat treatment and subsequent use of small dies made from plain carbon tool steel. This paper deals with the study of various effects of prequench treatments on the properties of the subsequently hardened tool steel. It is recommended that, for maximum hardness, this steel be air-normalized from 1650 degrees Fahr., water-quenched from 1470 degrees Fahr., and tempered at 390 degrees Fahr. Oil-normalizing from 1650 degrees Fahr., water-quenching from 1380 degrees Fahr., and tempering at 390 degrees Fahr. yields an equally high hardness. For maximum body value, it is recommended that this steel be annealed from 1470 degrees Fahr., water-quenched from 1380 degrees Fahr., and tempered at 390 degrees Fahr. For a combination of high hardness and good body, oil-normalizing from 1560 degrees Fahr., followed by water-quenching from 1470 degrees Fahr. and tempering at 390 degrees Fahr., is to be recommended.

Effect on the Hardness of Normalizing the Steel Previously to Quenching and Tempering Treatments

1. Preparation of Specimens. A billet of plain carbon tool steel was cut into 1¾ inch squares, about ¼ inch thick, and the surfaces rough ground. Chemical analysis showed this steel to contain: 1.01 per cent carbon, 0.02 per cent phosphorus, 0.01 per cent sulphur, 0.13 per cent silicon, and 0.24 per cent manganese.

2. Program of Heat Treatments:

(a) Steel as received.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author is a member of the society. He is associated with the research laboratories of the General Electric Co., Schenectady, N. Y. Manuscript received June 5, 1930.

(b) Steel air-normalized after 15 minutes at 900 degrees Cent. (1650 degrees Fahr.).

(c) Steel air-normalized after 15 minutes at 800 degrees Cent. (1470 degrees Fahr.).

(d) Steel oil-normalized after 15 minutes at 900 degrees Cent. (1650 degrees Fahr.).

(e) Steel oil-normalized after 15 minutes at 800 degrees Cent. (1470 degrees Fahr.).

(f) Three samples from each of the preceding treatments were water quenched after being held 15 minutes at each of the following temperatures: 725, 740, 745, 750, and 800 degrees Cent. (1335, 1365, 1375, 1380, and 1470 degrees Fahr.).

(g) Rockwell C hardness was taken on each of the hardened samples.

(h) The hardened samples were tempered successively at 200, 250, and 300 degrees Cent. (390, 480 and 570 degrees Fahr.) for ½ hour and the hardness measured after each tempering treatment.

3. Hardness Tests and Derived Curves. Four to six hardness tests were made on each specimen, and the average of all the tests on the three specimens for each treatment was considered as the characteristic hardness for that treatment.

Fig. 1 shows the variation in hardness with the change in quenching temperature for each of five prequench treatments.

Fig. 2 shows the hardness of the specimens from the 750 and 800 degrees Cent. (1380 and 1470 degrees Fahr.) quenches after tempering at 200, 250, and 300 degrees Cent. (390, 480 and 570 degrees Fahr.). These values are plotted against the prequench treatments for convenience of comparison.

4. Conclusions

(a) From Fig. 1, it is evident that 750 degrees Cent. (1380 degrees Fahr.) is the lowest full hardening temperature. The maximum values obtained on the 745-degree Cent. (1375 degrees Fahr.) quench show 745 degrees Cent. to be on the edge of the full hardening range.

The tendency in the 800-degree Cent. (1470 degrees Fahr.) quench is toward a slightly greater hardness than for the 750-degree Cent. quench. Since 800 degrees Cent. (1470 degrees Fahr.) is just above the A_{cm} point for this steel, it may be that the excess cementite

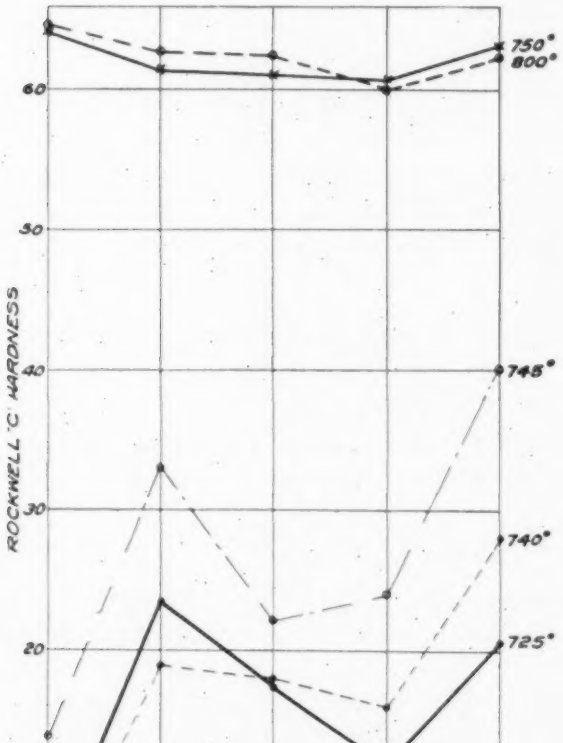


Fig. 1—Variation in Hardness with the Change in Quenching Temperature for Each of Five Prequench Treatments.

is nearer the critically dispersed state than after a 750-degree Cent. (1380 degrees Fahr.) quench and thus is slightly harder.

The 750 and 800 degree Cent. (1380 and 1470 degrees Fahr.) quenches indicate a tendency for the oil-normalized samples to be slightly harder as quenched than the air-normalized. The oil-normalized specimens have a smaller prequench grain size than the air-normalized and in a 15-minute heat, previous to quenching, it may be that the samples having a smaller prequench grain size come closer to structural equilibrium before quenching and yield a greater postquench hardness.

(b) In Fig. 2, the samples as tempered at 200 degrees Cent. (390 degrees Fahr.) show increases in hardness above that

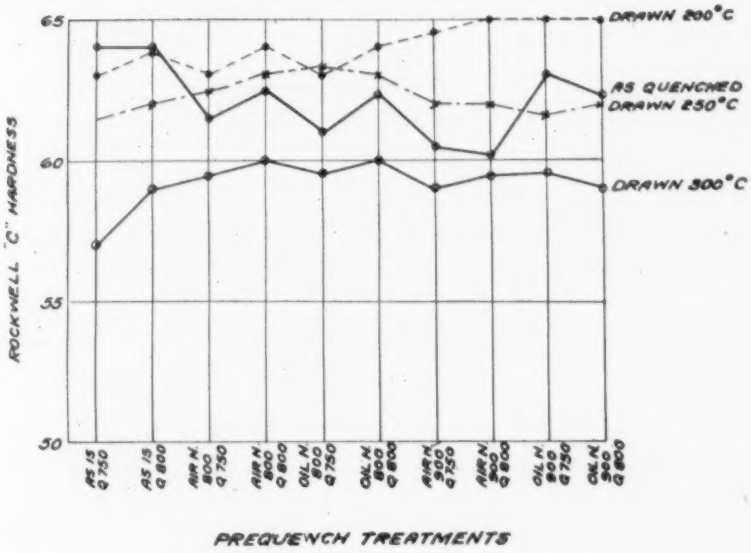


Fig. 2—Hardness of the Specimens from 1380 to 1470 Degrees Fahr. Tempered at 390, 480 and 570 Degrees Fahr. Values Plotted against Prequench Treatments.

of the quenched condition—the amount of increase depending on the prequench treatment.

It is well-known that tempering tends to decompose the austenite and martensite in quenched steels.

The "as received" condition shows a decrease in hardness after the 200 degrees Cent. temper. Thus the decomposition of the martensite is a greater factor than the decomposition of any retained austenite and, probably, there is little retained austenite in the "as received" condition quenched from 750 degrees Cent. (1380 degrees Fahr.). The "as received" quenched from 800 degrees Cent. (1470 degrees Fahr.) and tempered at 200 degrees Cent. indicates that just sufficient austenite was retained to counteract the softening effect of tempering martensite at 200 degrees Cent.

The air-normalized at 800 degrees Cent. and quenched at 750 degrees Cent., air-normalized at 800 degrees Cent. and quenched at 800 degrees Cent., oil-normalized at 800 degrees Cent. and quenched at 750 degrees Cent., oil-normalized at 800 degrees Cent. and quenched at 800 degrees Cent.,—all show the same amount of increase in hardness after tempering at 200 degrees Cent., and therefore all had about the same amount of retained austenite in the quenched state.

However, the samples, oil and air-normalized at 900 degrees Cent. (1650 degrees Fahr.), show a marked increase of 4 and 5 points in hardness after tempering and thus show a marked amount of austenitic retentivity in the quenched condition. The steels air-normalized from 900 degrees Cent. (1650 degrees Fahr.) give a softer steel in the quenched condition but show a C hardness of 65 when tempered at 200 degrees Cent. One might expect that air-normalizing at 900 degrees Cent. (1650 degrees Fahr.) before

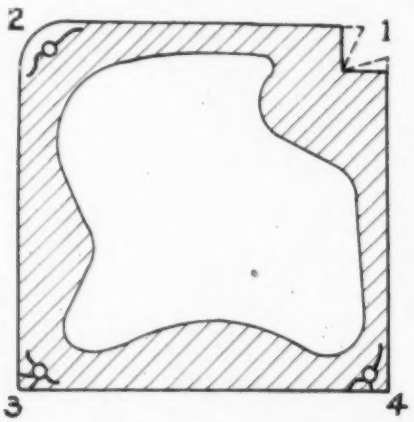


Fig. 3—Shape of Test Specimens Used.

water-quenching from 750 degrees Cent. (1380 degrees Fahr.) and tempering at 200 degrees Cent. would be the best treatment to obtain maximum hardness in such thin irregular sections as occur in some dies. The same treatment, but quenching from 800 degrees Cent., would be equally efficient for larger pieces of more regular shape.

(c) Tempering at 250 and 300 degrees Cent. resulted in a lowering of the hardness obtained by tempering at 200 degrees Cent. and shows that for maximum hardness the best of these tempering temperatures is 200 degrees Cent.

Effects of Nine Prequench Treatments on the Body of Tool Steel

It is well to define here what is meant by the term "body" as used in this research. By "body" is meant the ability of steel to go through repeated heating and quenching cycles without cracking. Thus, the number of such cycles that results in cracking may be used to measure qualitatively the body of the steel.

1. Preparation of Samples

The same 1.01 per cent carbon steel was used for these specimens. Pieces about 1¾ inches square and ¼ inch thick were cut from the bar and the surfaces rough ground. One corner was notched to a depth of ¼ inch and ⅛-inch holes were drilled about ⅛ inch from each of the remaining corners. One of these three corners was also filleted. Fig. 3 shows a view of the shape used. It is seen that a combination of three sets of strain conditions may be tested by such a specimen. These specimens are referred to as "die blanks."

2. Program of Treatments for Die Blanks

Nine prequench treatments were used and are listed here:

1. Steel as received.
2. Steel annealed 15 minutes at 750 degrees Cent. (1380 degrees Fahr.).
3. Steel annealed 15 minutes at 800 degrees Cent. (1470 degrees Fahr.).
4. Steel annealed 15 minutes at 850 degrees Cent. (1560 degrees Fahr.).
5. Steel annealed 15 minutes at 900 degrees Cent. (1650 degrees Fahr.).
6. Steel air-normalized after 15 minutes at 850 degrees Cent. (1560 degrees Fahr.).
7. Steel oil-normalized after 15 minutes at 850 degrees Cent. (1560 degrees Fahr.).
8. Steel air-normalized after 15 minutes at 900 degrees Cent. (1650 degrees Fahr.).
9. Steel oil-normalized after 15 minutes at 900 degrees Cent. (1650 degrees Fahr.).

In graphs of Figs. 4, 5, 6 and 7 these treatments are listed on the horizontal axes according to their numbers in the above list. Three samples from each prequench treatment were repeatedly quenched from each of the temperatures 750, 800, 850, and 900 degrees Cent. (1380, 1470, 1560, and 1650 degrees Fahr.) until all four corners of each specimen showed failure. For heating, the cold samples were placed in the furnace at the above indicated temperatures. Cracks visible under a magnification of × 10 were

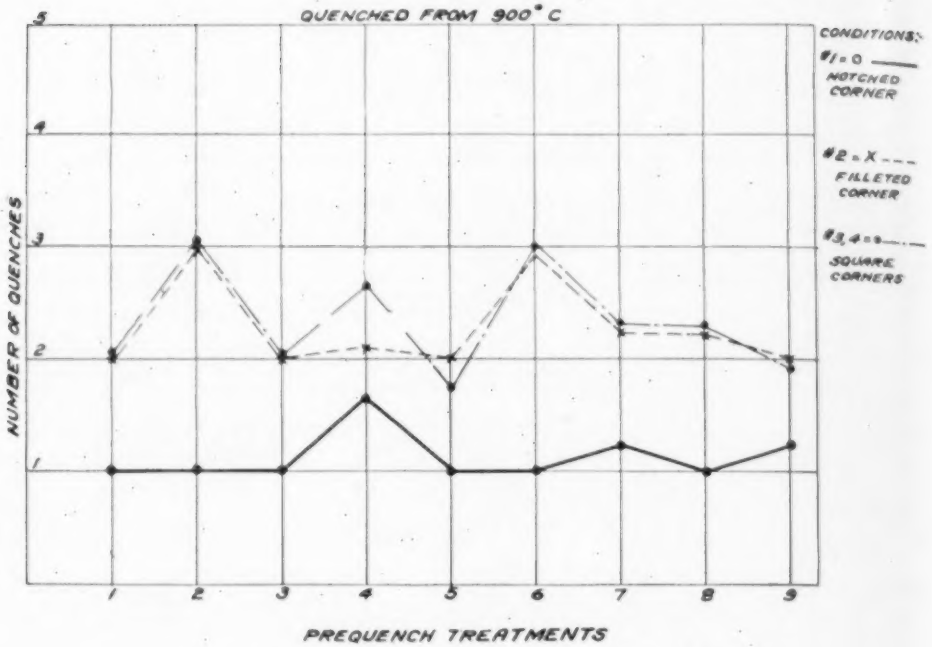


Fig. 4—Steel Quenched from 1650 Degrees Fahr.

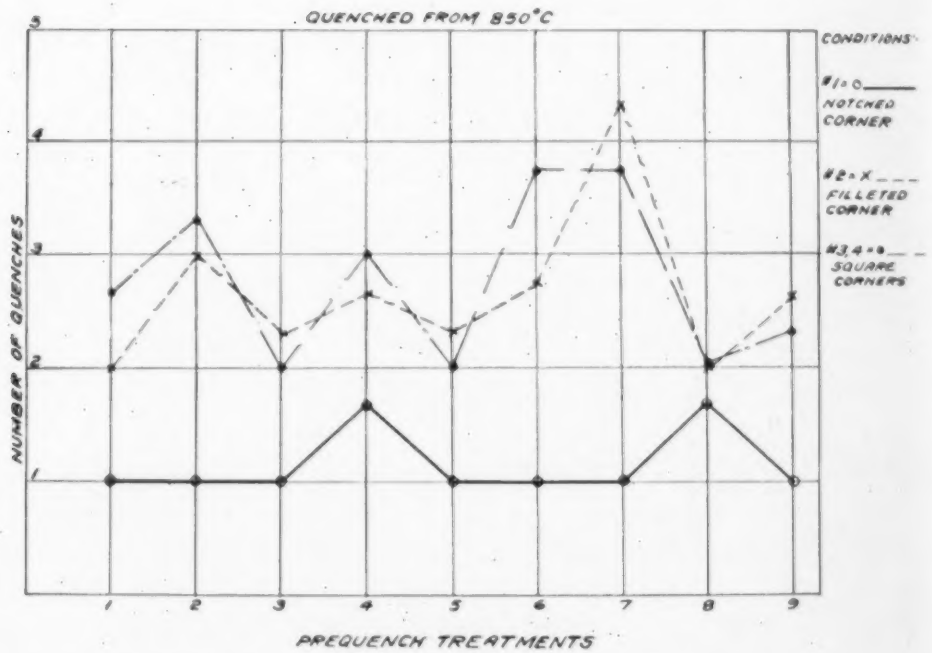


Fig. 5—Steel Quenched from 1560 Degrees Fahr.

obvious failures. In some cases with the notched corner, when cracks did not develop, visible distortion was regarded as failure.

3. *Results as Shown in Graphs*

The number of quenches required for the failure of each corner—at each of the four quenching temperatures as following the nine prequench treatments—was averaged and this average plotted against the prequench treatment. This average number of quenches may be a whole or fractional number and represents qualitatively the "body" of the steel as affected by the shape of the specimen and the nature of the prequench treatment. Thus, Figs. 4, 5, 6, and 7 show the effect of prequench treatment and corner conditions on the body of this steel when quenched repeatedly from 900, 850, 800, and 750 degrees Cent. (1650, 1560, 1470, and 1380 degrees Fahr.) into water.

4. *Conclusions from Graphs*

(a) Steel quenched from 900 degrees Cent. (1650 degrees Fahr.) and shown in Fig. 4.

It is at once apparent that heating to 900 degrees Cent. (1650 degrees Fahr.) practically destroys all effect of prequench treatment and the body for each corner condition is about constant. However, the notched corner failed uniformly at a lower body value than did the drilled corners.

(b) Steel quenched from 850 degrees Cent. (1560 degrees Fahr.) and shown in Fig. 5.

The notched corner failed uniformly at a low body value for all prequench treatments. The body of the drilled corners seems to be somewhat higher than that of the 900 degrees Cent. (1650 degrees Fahr.) quench. There is, generally, no appreciable difference in body between the filleted and the square cut drilled corners. The drilled corners do show to a small degree an optimum body condition for oil normalizing from 850 degrees Cent. (1560 degrees Fahr.) before quenching in water from 850 degrees Cent.

(c) Steel quenched from 800 degrees Cent. (1470 degrees Fahr.) and shown in Fig. 6.

With the lowering in quenching temperature, a greater effect of prequench treatments on body is at once noticeable. For the

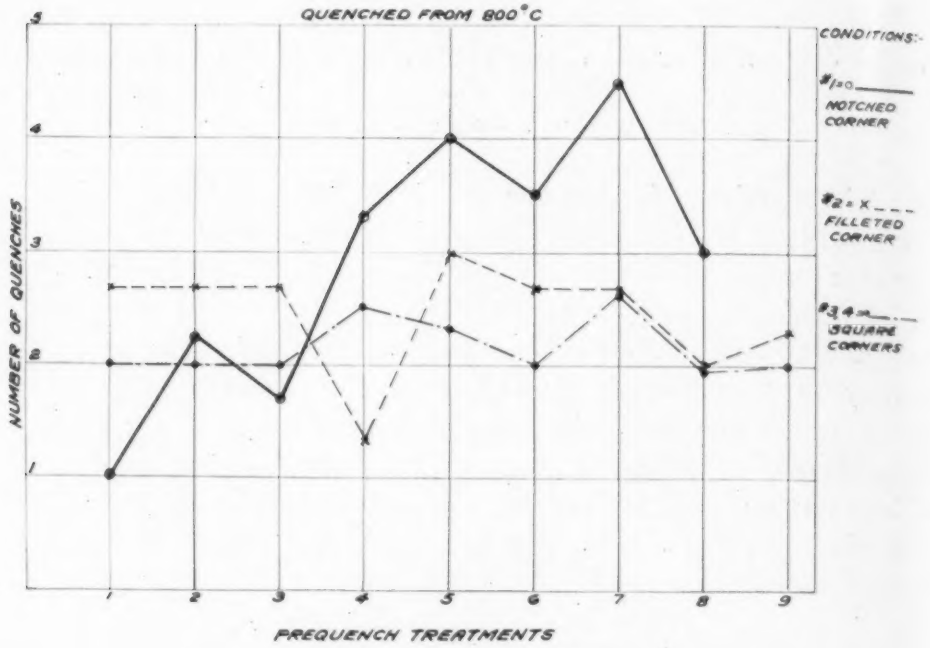


Fig. 6—Steel Quenched from 1470 Degrees Fahr.

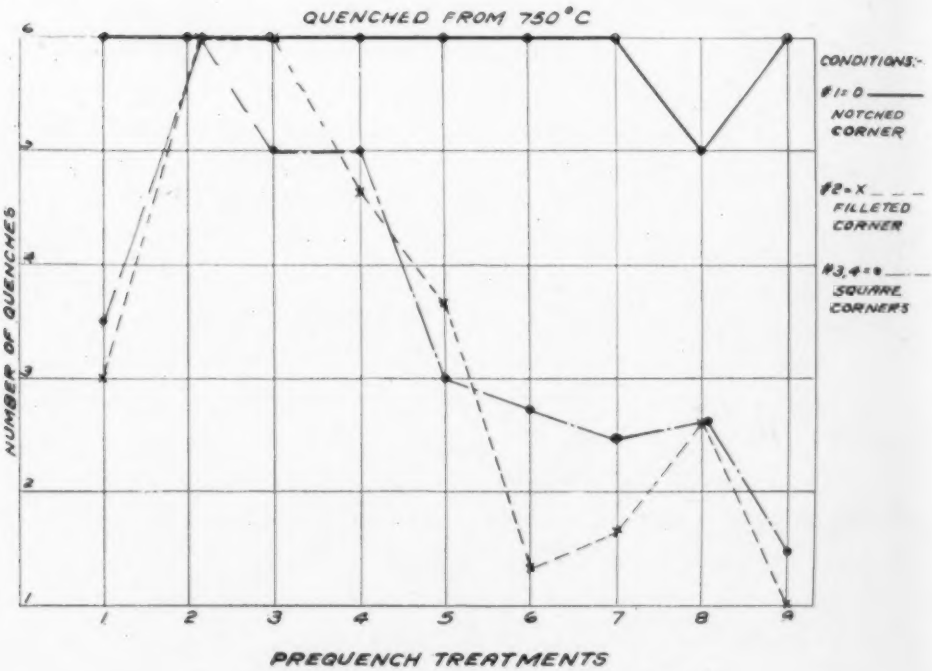


Fig. 7—Steel Quenched from 1380 Degrees Fahr.

prequench treatments consisting of the steel in the "as received" condition, the annealed from 750 degrees Cent. (1380 degrees Fahr.) and the annealed from 800 degrees Cent. (1470 degrees Fahr.) conditions, the notched corner has lower body value than the drilled corners. However, for all other prequench treatments consisting of the higher anneals and the normalized conditions, the notched corner has a markedly higher body than have the square and filleted drilled corners.

Apparently the 850 degrees Cent. (1560 degrees Fahr.) oil normalizing gives the optimum body after water-quenching from 800 degrees Cent. (1470 degrees Fahr.). It is to be noted that all the steel specimens quenched in water from 800, 850, 900 degrees Cent. (1470, 1560, 1650 degrees Fahr.) failed in body by actually cracking after the number of quenches as shown on the graphs.

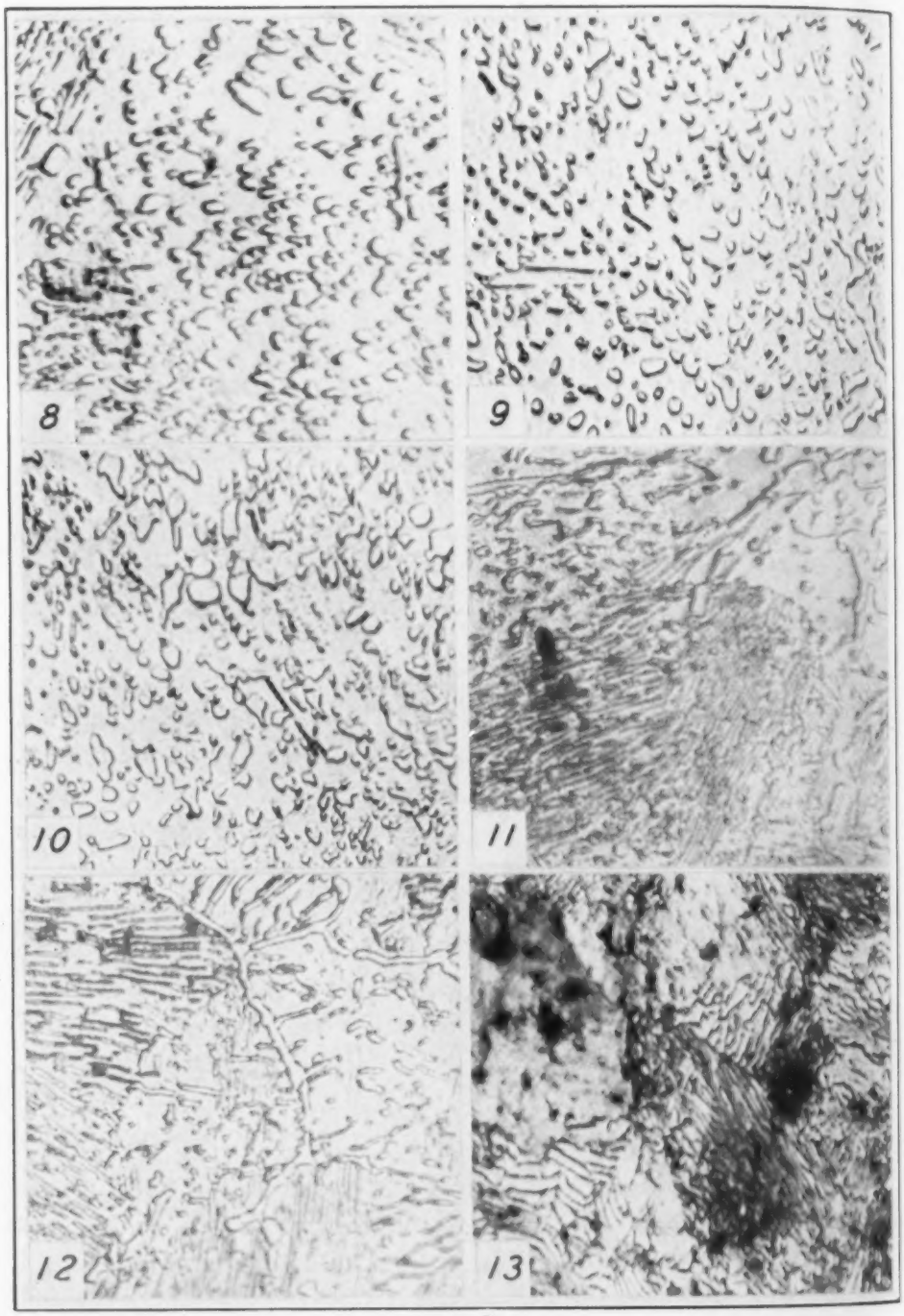
(d) Steel quenched from 750 degrees Cent. (1380 degrees Fahr.) and shown in Fig. 7.

It is at once apparent that the notched corner failed uniformly at a higher body value than did the drilled corners. With the exception of the 900 degrees Cent. (1650 degrees Fahr.) air-normalizing prequench treatment, the failure of the notched corner was not by cracking but by a noticeable dimensional distortion at the corner. The three drilled corners, by cracking at a lower body value, appeared to afford some relief and allowed the hoop tension to slightly close the notched corner.

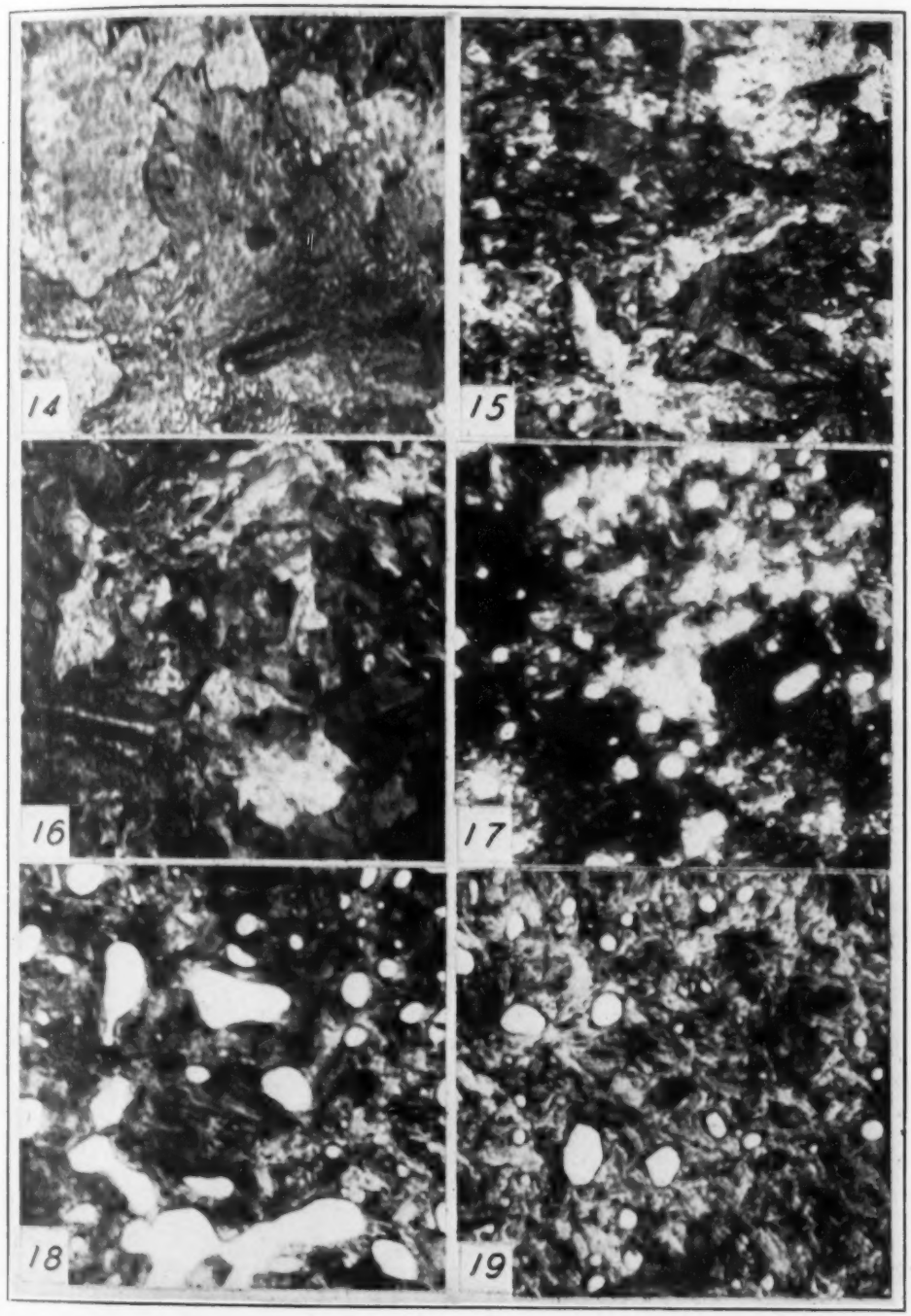
For the 750 degrees Cent. (1380 degrees Fahr.) water-quench the best body conditions result from the 750 and 800 degrees Cent. (1380 and 1470 degrees Fahr.) anneals as prequench treatments. The normalizing prequench treatments give lower body values for the 750 degrees Cent. (1380 degrees Fahr.) water-quench.

5. *Distribution of Stress in One Specimen*

One of the samples, oil-normalized from 900 degrees Cent. (1650 degrees Fahr.) before water-quenching from 750 degrees Cent., (1380 degrees Fahr.) showed definite variations in thickness after 10 quenches. Fig. 3 is a sketch of this sample. The shaded area is thin and the unshaded is thicker as tested by micrometers. The drilled corners cracked as shown while the notched corner, No. 1, tended to close. It seems probable that the thin areas indicate a rough hoop of tension with the center of the piece in compres-



Figs. 8 to 13—Structures After Prequench Treatments.
Fig. 8—As Received. Fig. 9—After Annealing at 1380°F. Fig. 10—After Annealing at 1470°F. Fig. 11—After Annealing at 1560°F. Fig. 12—After Annealing at 1650°F. Fig. 13—After Air-Normalizing at 1560°F. All Specimens Etched in 3% Nital. × 750.



Figs. 14 to 19—Structures After Prequench Treatments.
Fig. 14—After Air-Normalizing at 1650°F. Fig. 15—After Oil-Normalizing at 1560°F. Fig. 16—After Oil-Normalizing at 1650°F. Fig. 17—As Received after Quenching from 1380°F. Fig. 18—Annealed at 1380°F. and Quenched from 1380°F. Fig 19—Annealed at 1470°F. and Quenched at 1380° F. 3% Nital Etch. First 3 Pictures, × 750. Last 3 Pictures, × 1500.

sion and the outside of the filleted corner in relief as a result of cracking.

Structures as Affected by the Prequench Treatments

1. Structures after Prequench Treatments

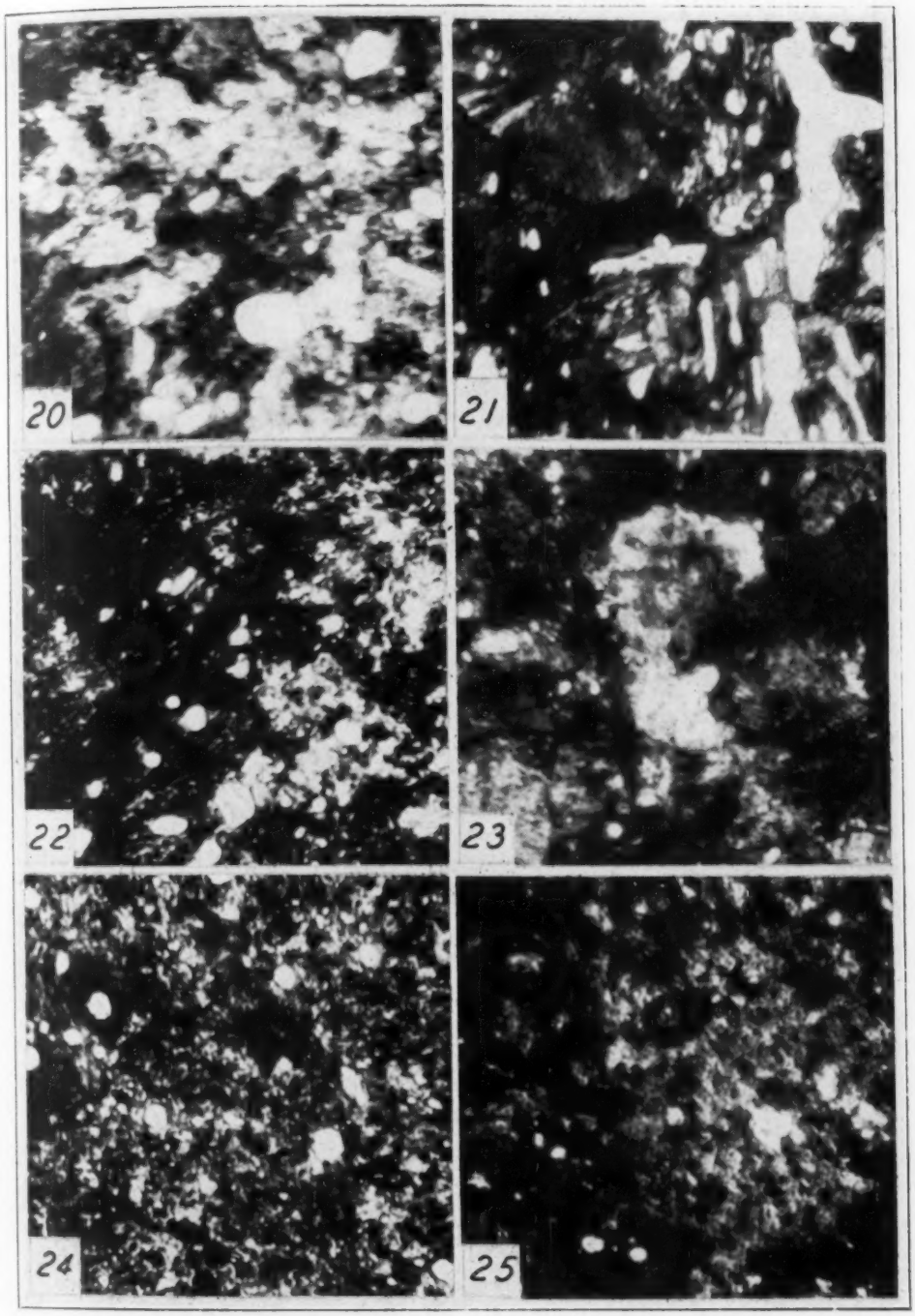
The samples were polished, etched with 3 per cent nital and representative photomicrographs were taken at a magnification of 750 diameters.

Fig. 8 is of the steel in the "as received" condition and shows non-uniformity in structure, the carbide being present in both spheroidal and lamellar conditions. Fig. 9 shows the structure after annealing at 750 degrees Cent. (1380 degrees Fahr.). The spheroidizing of the carbide is more complete. In Fig. 10 is shown the spheroidized structure obtained from annealing at 800 degrees Cent. (1470 degrees Fahr.). Possibly there is a larger number of very small carbide particles in the 800 degrees Cent. (1470 degrees Fahr.) anneal. Fig. 11 is of the steel annealed at 850 degrees Cent. (1560 degrees Fahr.) and shows a coarse lamellar pearlite lacking any definite carbide network. Fig. 12 shows the structure after annealing at 900 degrees Cent. (1650 degrees Fahr.). This is a coarse abnormal pearlite with a marked network of carbide.

In Fig. 13 is shown the structure of the steel after air-normalizing from 850 degrees Cent. (1560 degrees Fahr.) which resulted in the excess cementite becoming partly spheroidized. Fig. 14 shows the structure of the steel, air-normalized from 900 degrees Cent. (1650 degrees Fahr.), to be largely sorbitic pearlite. Fig. 15 is of the steel oil-normalized from 850 degrees Cent. (1560 degrees Fahr.) and shows a mixture of sorbite and troostite with fine excess cementite particles well distributed. Fig. 16 shows the steel after oil-normalizing from 900 degrees Cent. (1650 degrees Fahr.). This structure is again a mixture of troostite and sorbite, but the excess cementite is of so fine a particle size as to be imperceptible.

2. Structures after One Quench from 750 degrees Cent. (1380 degrees Fahr.)

The prequench structures would seemingly have a greater effect on the lowest temperature quenching treatment. Therefore, structures are shown of all nine prequench treatments after a succeeding single water-quench from 750 degrees Cent. (1380 degrees Fahr.).



Figs. 20 to 25—Structures After Prequench Treatments.
Fig. 20—Annealed 1560°F. followed by 1380°F. Quench. Fig. 21—Annealed 1650°F., Quenched 1380°F. Fig. 22—Air Normalized from 1560°F. and Quenched from 1380°F. Fig. 23—Air Normalized from 1650°F. and Quenched from 1380°F. Fig. 24—Oil Normalized from 1560°F. and Quenched from 1380°F. Fig. 25—Oil Normalized from 1650°F. and Quenched from 1380°F. All Samples Etched 3% Nital. × 1500.

These photomicrographs were taken at a magnification of × 1500 in order to better show the way in which the excess carbide occurs. Thus the matrix may be a little out of focus and has been printed a little darker in order to distinguish the carbide clearly.

Fig. 17 shows the "as received" condition after quenching. The cementite particles are of all sizes and shapes in a non-uniform matrix of troostitic martensite. The "as received" does not seem suitable for immediate quenching treatments. Fig. 18 shows the 750 degree Cent. (1380 degrees Fahr.) annealed sample after quenching. The excess cementite is all spheroidized, but shows a tendency to form in what may have been a preceding austenite grain boundary. It is also possible that this network tendency results from the non-uniform "as received" steel unaffected by 750 degrees Cent. (1380 degrees Fahr.) annealing and quenching, therefore dating back to the austenite grain boundaries occurring during the fabrication of the steel. The matrix is fine grained martensite. Fig. 19 shows the steel after annealing at 800 degrees Cent. (1470 degrees Fahr.) and quenching at 750 degrees Cent. (1380 degrees Fahr.). The carbide particles are small and uniformly distributed in a bed of martensite. This would seem to be an excellent condition and did show good body conditions. Fig. 20 of the 850 degrees Cent. (1560 degrees Fahr.) anneal, followed by 750 degrees Cent. (1380 degrees Fahr.) quenching, shows large irregular cementite particles due probably to the prequench coarse lamellar pearlite. The matrix is a non-uniform troostitic martensite.

Fig. 21 is very interesting. It shows the steel as quenched from 750 degrees Cent. (1380 degrees Fahr.) after annealing at 900 degrees Cent. (1650 degrees Fahr.). The matrix is again troostitic martensite. However, the excess carbide network from the prequench high annealing treatment remains nearly unbroken. Also some of the very coarse lamellar cementite of the prequench pearlite still remains after the quenching treatment.

Figs. 22 and 24 show the structures resulting from quenching the 850 degrees Cent. (1560 degrees Fahr.) air and oil-normalized structures. The excess cementite particles are distributed in a field of troostitic martensite which is more uniform in the case of the oil-normalized sample. It is to be expected that normalizing from a higher temperature (within the normalizing range) would result in finer carbide particles after quenching. This is shown by Figs.

23 and 25 of the steel for prequench air and oil-normalizing from 900 degrees Cent. (1650 degrees Fahr.). The 900 degrees Cent. normalized samples, because of finer cementite particles, should be somewhat harder, after quenching and drawing back the retained austenite, than is the case for the 800 degrees Cent. (1470 degrees Fahr.) normalizing. This is shown by the curves in Fig. 2.

General Conclusions

1. If articles of tool steel are being heat treated on a large scale, requiring as short a time at temperature as possible, the prequench condition and recommended prequench heat treatments become factors of some importance.

2. For maximum hardness this steel should be air-normalized from 900 degrees Cent. (1650 degrees Fahr.), quenched from 800 degrees Cent. (1470 degrees Fahr.) and tempered at 200 degrees Cent. Oil-normalizing from 900 degrees Cent., water-quenching from 750 degrees Cent. (1380 degrees Fahr.) and tempering at 200 degrees Cent. yields an equally high hardness.

3. For maximum body value this steel should be annealed from 750 or 800 degrees Cent. (1380 or 1470 degrees Fahr.) and water-quenching from 750 degrees Cent.

4. For a combination of high hardness and good body, oil-normalizing from 850 degrees Cent. (1560 degrees Fahr.) followed by water-quenching from 800 degrees Cent. (1470 degrees Fahr.) is recommended.

5. From the consideration of both hardness and body, the steel in the "as received" condition should not be quenched without some prequench treatment.

DISCUSSION

J. V. Emmons:[1] Mr. Harrington is to be congratulated upon the very high quality of his microscopic work. It illustrates his structures very admirably. The question of body in tool steels has always been a very interesting one. I recall reading a short time ago in the *Transactions* of the British Iron and Steel Institute, that the question was put to a famous Swedish metallurgist as to the nature of the body in Swedish steels, which was supposed to make them better than other steels. The Swedish metallurgist replied, "My grandmother told me not to tell such things." Mr. Harrington is really making an effort to get the "body" of tool steel out of the class of things which our grandmother

[1]Metallurgist, Cleveland Twist Drill Co., Cleveland.

said we should not tell. That is, to get it down to an actual basis of recorded scientific facts. I am not prepared to offer definite suggestions as to further methods of procedure in regard to this alleged body. It is, however, going to tie up to measurable properties of the materials in question.

G. V. LUERSSEN:[2] Mr. Harrington has undoubtedly touched upon a very interesting line of research on carbon steels. I have had occasion to work along in a similar line in connection with certain variables in the steel itself, although the influence of preheat treatment was not covered as completely as in the present paper. Mr. Harrington is to be congratulated upon the thoroughness with which he has treated this part of the work.

However he aptly stated at the conclusion of his talk that the results of his paper apply only to one particular steel. This is a very true statement, since important variations may exist in the behavior of two samples of tool steel of the same analysis given the same heat treatment. This fact was brought out in two papers on the subject of inherent hardenability presented before this Society last year, one by B. F. Shepherd and one by the present speaker, in which it was shown that the inherent characteristics often far outshadow variations in heat treatment. Following the presentation of these papers I had occasion to check up some of these steels on the basis of the test used by Maurer and Haufe, who estimated body figures as the number of requenchings possible upon prismatic specimens without cracking. In applying this test to carbon steels having different inherent hardness penetration, we found that the deep hardening steels having a comparatively narrow quenching range cracked after just a few repeated quenchings, while the shallower hardening steels with a wide quenching range stood a large number of requenchings. The deep hardening specimens actually became concave on the faces, while the shallow hardening specimens became convex. All were of the same analysis and were given the same treatments, differing only in inherent hardening properties.

It is obviously important therefore in work of this sort to carefully define the inherent characteristics of the steel under consideration. This statement does not in any way detract from the valuable work presented by Mr. Harrington, but is made rather in the hope that he will continue his work to include steels of different characteristics.

S. L. HOYT:[3] The method which Mr. Harrington has used to determine the body of steel qualitatively has proven to be most valuable in research work. I was visiting in Sheffield, England, a few years back and, being one afternoon with two leading metallurgists, I asked them, "After all, what do you mean by body?" One of them told me that steel with body was steel which could be repeatedly hardened without distorting considerably or without cracking. It remained for Maurer at the Krupp Laboratory to develop a method which was capable of giving reproducible results. Maurer's sample was ¾ of an inch square by 8 inches long. In the research laboratory at Schenectady a number of us have used this particular method for studying body in steel and the results have been gratifying. But one of the most interesting points here is, after all, what is responsible for body. The answer seems to be associated very definitely

[2]Metallurgical Department, Carpenter Steel Co., Reading, Pa.

[3]Research metallurgist, General Electric Co., Schenectady, N. Y.

with melting stock. That does not agree with comments made early this afternoon, but insofar as the crucible process with Swedish iron as melting stock is concerned, the body of the finished steel depends on the method of making the Swedish iron. The body of the steel improves as the size of the charge, that is the Swedish iron, is reduced and as the time of working this charge is increased. Personally I should like to see either Dr. Harrington or somebody else who is working definitely in this field secure some steels which are known by practical experience to possess this property of body to an outstanding degree and then to make a more thorough study of its structure. It means going beyond what has already been reported here today, paying particular attention to the nature and character of the foreign inclusions in the iron. If you will recall for a moment what I said about the body of crucible steel depending upon the melting stock it seems quite likely that the body therefore will depend at least partially upon the foreign inclusions which are present, their number, and dispersion. I will not go into that. I wish merely to offer this suggestion that more thought be put upon that particular point in connection with body. One steel that we got in Schenectady (I do not know who sold it to us) showed very low body figures, and in correspondence with Dr. Maurer, he confirmed my conclusion that the steel was extremely poor in quality. The microscope revealed at once that there was a very definite difference between this steel and a normal steel of the high carbon tool steel type. So it would seem to be very much worth while, in the future, to pay more attention to the microstructure of the annealed steel. I believe that kind of study will lead us definitely forward in the study of body.

A. H. d'Arcambal:[1] Just a few comments in connection with this excellent paper by Dr. Harrington. In the first place I believe that a word of warning should be issued in connection with using a hardening temperature of 1380 degrees Fahr. for 1 per cent carbon tool steel. In commercial practice this is too low a temperature. Quenching in the critical range, this type of steel will not harden satisfactorily and quite often your work will crack when quenched from such a temperature. In regard to the hardness values obtained, we find no difficulty whatever in obtaining a Rockwell hardness of C65 to 67 on this type of steel, hardening it as received from the steel mill, that is, a structure so annealed as to have at least 75 per cent of the structure completely spheroidized.

Author's Closure

I wish to thank Mr. Emmons, Mr. Luerssen, Dr. Hoyt, and Mr. d'Arcambal for their comments regarding the research reported in this paper. The fine photomicrographs, referred to by Mr. Emmons, were taken by Mr. L. L. Wyman of the General Electric Research Laboratory.

Mr. Luerssen made several important points regarding steels of different inherent hardenability characteristics. Since the paper deals with a single steel it may be of some interest to continue this work on various steels having inherent differences. It seems probable, given a steel of certain hardenability characteristics, that the proper prequench treatment may be used to increase the body of deep hardening steels or to somewhat increase the depth of hardness

[1]Consulting metallurgist, Pratt and Whitney Co., Hartford, Conn.

in high body, shallow-hardening steels—depending upon what may be required from the steel in actual usage.

Dr. Hoyt and Mr. Luerssen have mentioned the work of Dr. Maurer who used a single specimen shape throughout his excellent research on body. Thus it would seem that one of the variables affecting body automatically became a constant for the purposes of his research. The varying corner conditions, as found by the writer in his die-shaped specimens, indicate that the shape of the specimen is one of the factors determining the body of a steel.

It is perhaps fortunate that Mr. d'Arcambal suggested for commercial work a temperature slightly higher than that of 1380 degrees Fahr. mentioned by the writer in a suggested heat treatment. By interpolation of the data from the 1380 and 1470 degrees Fahr. quenches it is quite probable that temperatures of 1400 and 1420 degrees Fahr. would yield results similar to those of the 1380 degrees Fahr. quench.

In conclusion it would seem at this time that "body" in steel may be determined by the following factors:

1. Inherent hardenability characteristics as determined or affected by:
    a. Melting operations in producing the steel.
    b. Oxygen and other solutes.
    c. Foreign inclusions.
2. Heat treatments.
3. Shape of specimens with resultant variations of stress conditions of hardened steel.

STRESSES AND CRACKS IN HARDENED AND GROUND STEEL

By Gerald R. Brophy

Abstract

The work described in this paper deals with stresses introduced in hard steel surfaces by grinding and the detection and classification of these stresses.

Pickling causes the stressed areas to crack and acids of different concentration pick out the high and low stresses. The time in the bath is also a measure of stress. Low stresses require a stronger acid or a longer time in a given acid than do high stresses. The influence of heat treatment is also discussed.

THE subject of stress distribution in steel, as the result of quenching, has been studied by several investigators, notably Messrs Heyn,[1] Scott[2] and Hoyt,[3] and means have been devised for measuring the stresses. The stresses studied have been more or less uniform in respect to the surface of the steel, while the stresses dealt with in this paper are nonuniform in distribution and intensity, varying from point to point on the surface and up to the breaking point of the steel, and have been introduced in the steel by grinding.

The means for detecting the stresses, if they had not already been evidenced by cracking, was to pickle in various acids for varying lengths of time. Some interesting observations are also made on the question of the so-called embrittlement of steel by hydrogen during pickling.

A large amount of research has been done on this embrittle-

[1]E. Heyn, "Theorie der Eisen Kohlenstoff Legierungen," 1924.

[2]Howard Scott, "Origin of Quenching Cracks", Scientific Paper No. 513, U. S. Bureau of Standards.

[3]S. L. Hoyt, "Stresses in Quenched and Tempered Steel", Transactions, American Society for Steel Treating, Vol. 11, 1927, p. 509.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, Gerald R. Brophy, member of the society, is connected with the research laboratories of the General Electric Co., Schenectady, N. Y. Manuscript received May 28, 1930.

ment, and it has been shown by Fuller,[4] Parr[5] and others that when highly stressed steel, and especially hard steel, is pickled in either HCl or H_2SO_4 or when plated in an acid bath, it becomes brittle and frequently cracks. The embrittlement may be eliminated by aging at room temperature or by heating, but of course the weakening and embrittlement resulting from the formation of cracks is permanent.

The reasons for the embrittlement and cracking of steel under these conditions is connected with the passage of atomic hydrogen through the steel. Williams and Homerberg[6] suggest that the atomic hydrogen reduces iron oxide which may be contained in the steel forming water between the grain boundaries, and that the stress resulting, added to the stress already present, causes the steel in some cases to crack.

Fuller believes however that atomic hydrogen diffusing through steel arrives at a discontinuity and there changes to molecular hydrogen. It is then incapable of diffusing further. The atomic hydrogen continues to arrive and change, and therefore builds up a pressure sufficient to crack the steel.

If these ideas are correct, then an acid like HNO_3, which does not form hydrogen when steel is dissolved in it, ought not to cause cracking. It was found in this work that this apparently is true and therefore HNO_3 was used as a preliminary pickle to indicate the presence or absence of cracks previous to pickling in other acids.

No attempt has been made to measure the stresses quantitatively, but differences of degrees are indicated by the methods employed.

A frequent cause of failure of a hardened and ground steel surface is a network of practically invisible cracks which developes during or immediately after grinding. An example of this is shown in Fig. 1.

The literature on grinding gives a large amount of valuable information on the effects of speeds, size and grade of grinding

[4]T. S. Fuller, "Prevention of Brittleness in Electroplated Springs", *Transactions*, American Electro Chemical Society, Vol. 32, 1917.

[5]F. W. Parr, "Embrittling Action of Sodium Hydroxide in Soft Steel", University of Illinois Bulletin, No. 94, 1917.

[6]R. S. Williams and V. O. Homerberg, "Why Caustic Solutions Make Steel Brittle", *Chemical and Metallurgical Engineering*, Vol. 30.

[7]E. G. Mahin, Notre Dame University, *Proceedings*, Academy of Science, Vol. 37, 1927.

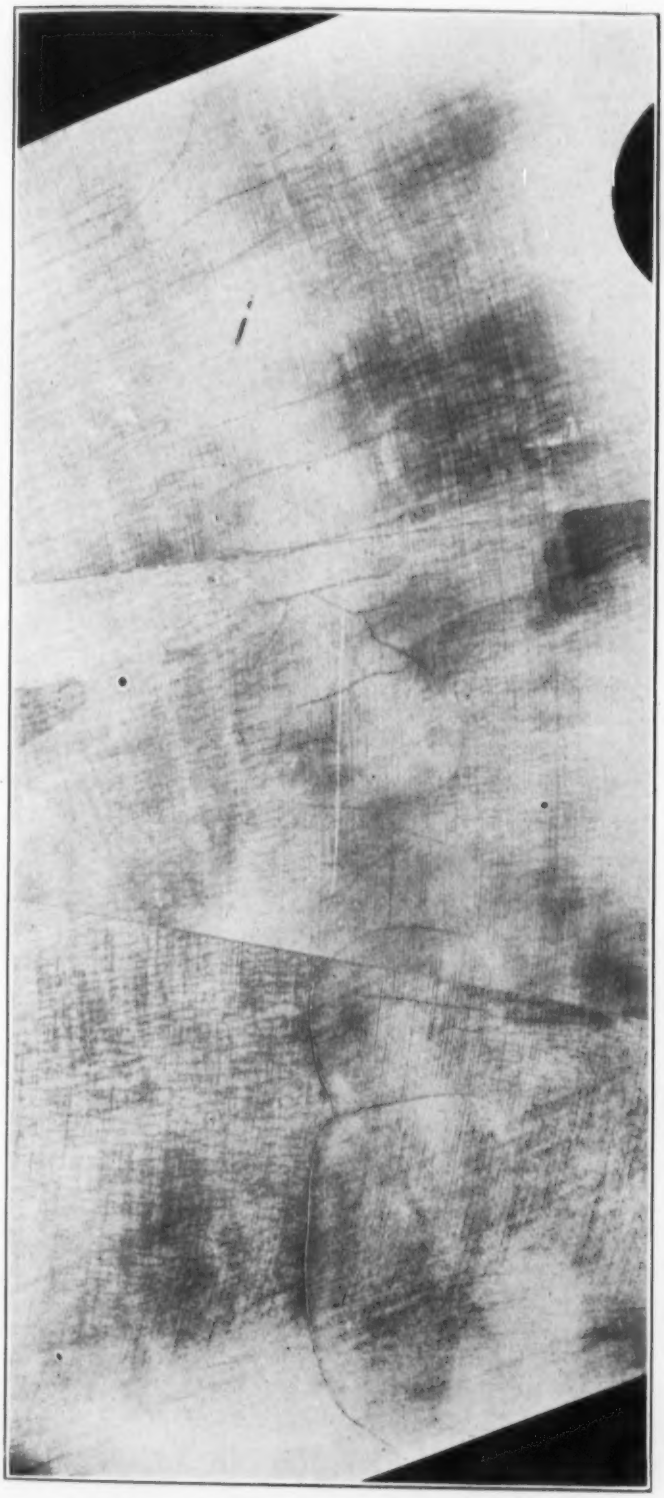


Fig. 1—Grinding Cracks Formed During Grinding.

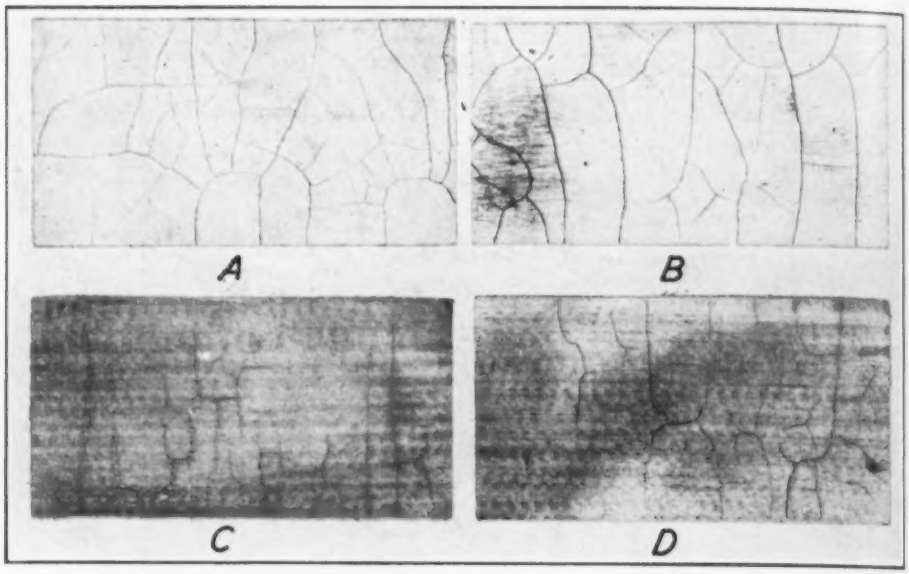


Fig. 1A—Dirty Steel—Showing Grinding Cracks 1445 Degrees Fahr. (785 Degrees Cent.) Quench—390 Degrees Fahr. (200 Degrees Cent.) Temper. Rockwell C-62 Top Unetched. Bottom Etched Slightly.

wheels and other strictly mechanical factors, but the effect of heat treatment, and the physical condition of the steel with respect to solid nonmetallic inclusions is but suggestively touched upon. It is with these two phases that the present report is concerned.

Influence of Solid Nonmetallics

Two steels of about the same composition (0.89 and 0.91 per cent C) were selected. One had a high dirt content and the other was relatively clean. Samples 1x¼x1½ inches were water-quenched from 1445 degrees Fahr. (785 degrees Cent.) and tempered at 390 degrees Fahr. (200 degrees Cent.) for 1 hour. The Rockwell hardness number was C 62. These were then dry-ground using a (38-46H) Norton wheel and removing 0.030 inch from each face in 0.003 inch cuts. The dirty steel was seriously cracked (Fig. 1-A) while the clean steel was not.

The cracks could be seen growing after the pieces were removed from the magnetic chuck.

Influence of Heat Treatment

Samples of clean 1.16 per cent C steel were quenched from three different temperatures namely: 1400, 1445 and 1520 degrees

Fahr. (760, 785 and 825 degrees Cent.). The duplicate set was tempered at 390 degrees Fahr. (200 degrees Cent.) for 1 hour. Before grinding, the samples were examined for visible cracks. The three samples quenched from 1520 degrees Fahr. (825 degrees Cent.) and untempered and one of the three tempered samples showed large straight quench cracks. All the samples were then pickled in 30 per cent HNO_3 and no additional cracks were found. When pickled 3 minutes in 30 per cent HCl no further cracks were found.

These were then ground as before and examined. The four samples quenched from 1520 degrees Fahr. (825 degrees Cent.) now showed a fine network of cracks visible between the large cracks, but none of the other samples were cracked. All were again pickled in HNO_3 and nothing further developed. Upon immersion in 15 to 20 per cent H_2SO_4 for 3 minutes, cracks developed as shown in Fig. 2. There is also a strong tendency for the surfaces to shell off in a layer about 1/16 inch thick.

An interesting observation was made on this series and was borne out in subsequent experiments. That is, one face is always more seriously cracked than the second and it is the first face ground which cracks. Frequently the second face ground would show no cracks. It is believed that the cracking on one face relieves at least some of the stress on the opposite face so that no cracking occurs there.

A second series of samples of the same steel was prepared by annealing at 1650 degrees Fahr. (900 degrees Cent.) to develop a large, coarse network of cementite and a like number were air cooled from 1650 degrees Fahr. (900 degrees Cent.) to break up any existing network. These were then quenched from 1445 degrees Fahr. (785 degrees Cent.), pickled in 15 per cent H_2SO_4 for 3 minutes and examined for cracks, but none were found.

This pickling was done principally to determine whether any cracks existed before grinding and also demonstrates quite conclusively that it is not necessarily dangerous to pickle uniformly hardened and unground steel, though undoubtedly temporary brittleness does result.

Grinding was done as before and again the samples were pickled for 3 minutes, but this time H_2SO_4 of 5, 15 and 30 per cent concentration was used. Fig. 3 shows the result. The samples containing a cementite network were all badly cracked and especially

730° C. 785° C. 825° C.

as quenched

Tempered at 200° C.

Fig. 2—1.16 Carbon Steel Showing Effect of Quenching Temperature on Occurrence of Grinding Cracks.

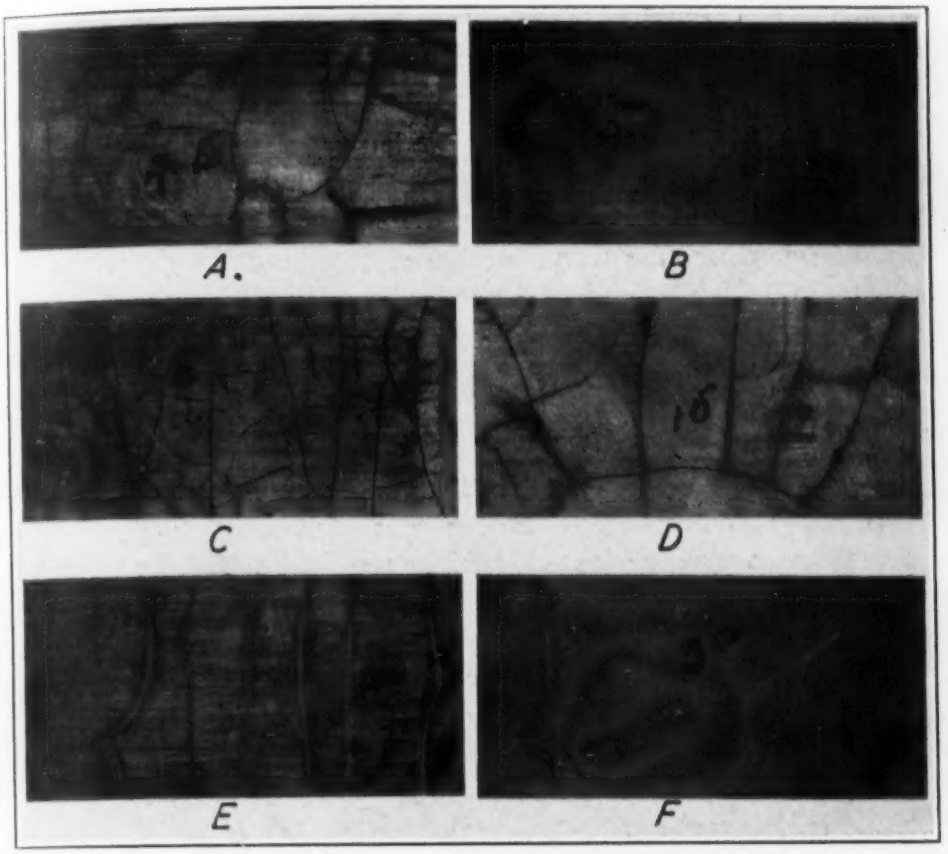


Fig. 3—Effect of Prequench Treatment and Acid Strength on Crack Development. Specimens A, C, E Annealed at 1650 Degrees Fahr. (900 Degrees Cent.) and Etched in 5, 15 and 30 Per Cent Sulphuric Acid Respectively.
Specimens B, D, F Normalized at 1650 Degrees Fahr. (900 Degrees Cent.) and Etched in 5, 15 and 30 Per Cent Sulphuric Acid Respectively.

the ones pickled in 15 per cent acid. The normalized samples were not as badly cracked, but again the 15 per cent acid produced the worst cracking.

A duplicate set was tempered at 300 degrees Fahr. (150 degrees Cent.) for 1 hour previous to grinding. The pickling time had to be extended now considerably until the first cracks appeared. The character of the cracks has changed to some degree, but again the annealed samples are those that cracked and no sign of a crack was found in the normalized samples. Fig. 4 shows this group after 6 minutes pickling.

Fig. 5 shows a similar group which had been tempered at 480 degrees Fahr. (250 degrees Cent.) for 1 hour. The Rockwell hardness is now C-59. They were ground as before, but this time pickled

Fig. 2—1.16 Carbon Steel Showing Effect of Quenching Temperature on Occurrence of Grinding Cracks.

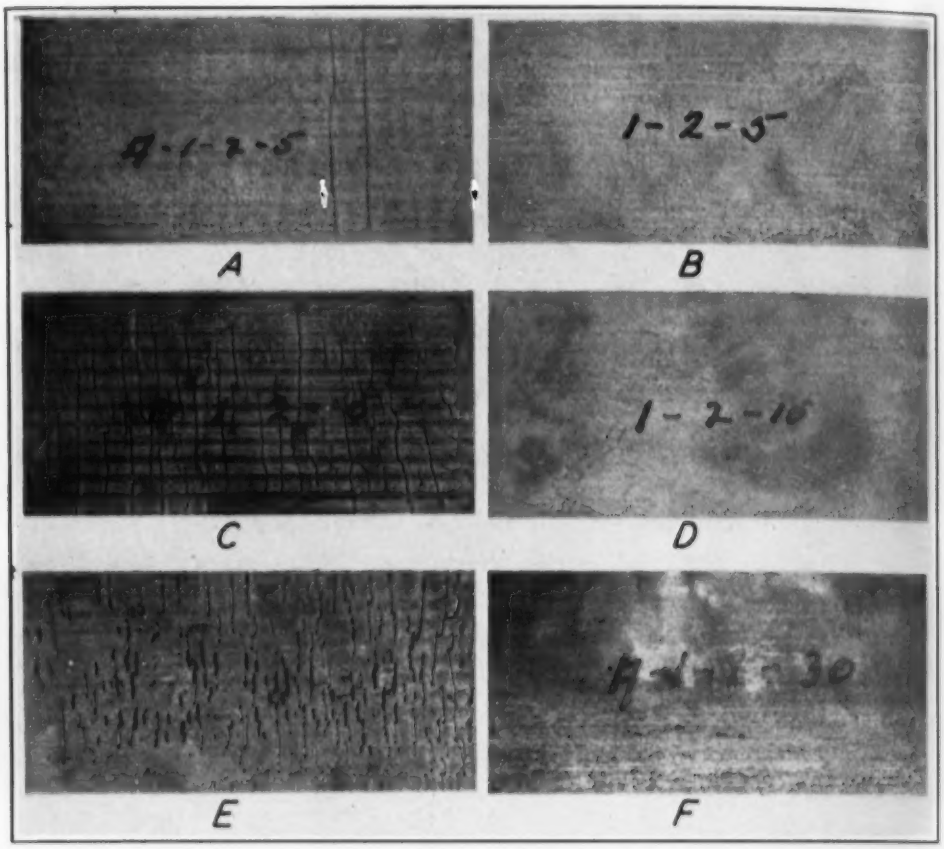


Fig. 4—Same as Fig. 3 but Tempered at 300 Degrees Fahr. (150 Degrees Cent.). Specimens Etched as in Fig. 3.

for 20 minutes and no cracking resulted in any of them.

A group of bars (Fig. 6) were quenched from 1445 degrees Fahr. (785 degrees Cent.) and tempered at 300 degrees Fahr. (150 degrees Cent.) for 1 hour and then ground, but after grinding were tempered again at 300 degrees Fahr. (150 degrees Cent.) for 2 hours. After pickling 20 minutes in 5 and 15 per cent H_2SO_4 no cracking resulted. This series is to be compared with the series of like treatment (Fig. 4) which were not tempered after grinding.

Influence of Time of Pickling

It was noticed early in these experiments that the length of time of pickling was a factor in the extent of cracking of the ground surfaces. The samples, shown in Fig. 2, were repickled in 15 per cent acid for 10 minutes and it was then found that a network of cracks apeared on all the samples that were quenched from 1520

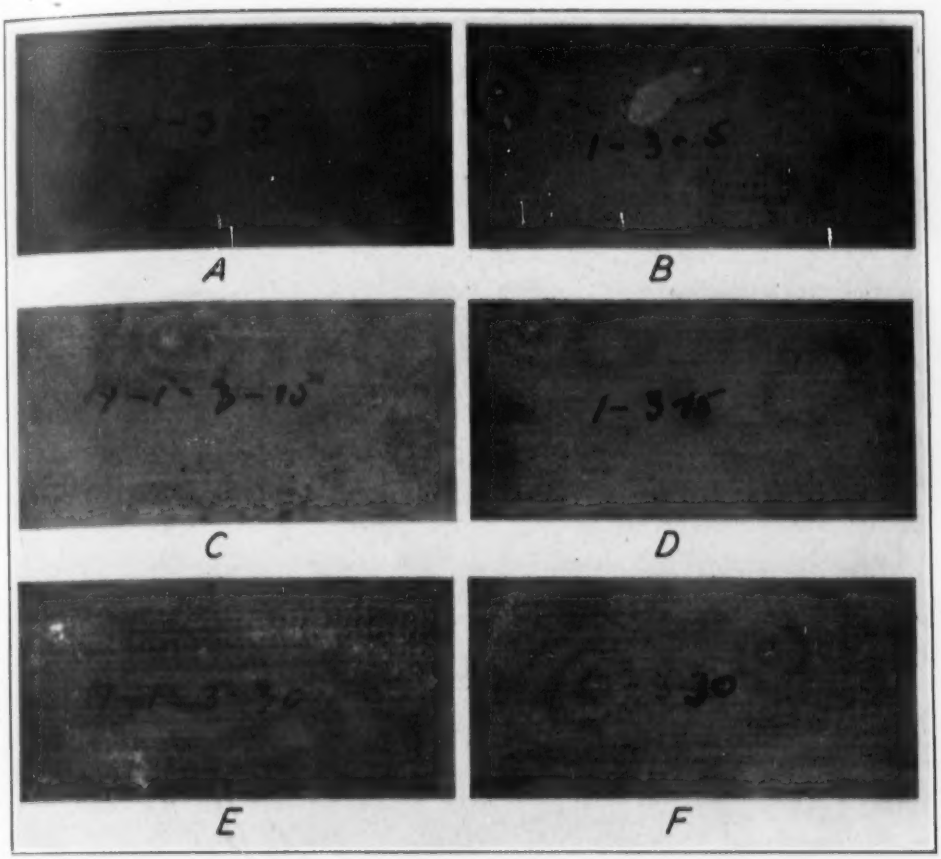


Fig. 5—Same as Fig. 3 but Tempered at 480 Degrees Fahr. (250 Degrees Cent.). Specimens Etched as in Fig. 3.

degrees Fahr. (825 degrees Cent.) and tempered, and on some of those quenched from 1445 degrees Fahr. (785 degrees Cent.). After an additional 10 minutes in the pickle all samples were cracked, except those quenched from 1380 degrees Fahr. (750 degrees Cent.) and tempered at 390 degrees Fahr. (200 degrees Cent.).

Effect of Acid Strength

Fig. 3 shows the influence of the strength of the acid on the number of cracks appearing. It can be noticed that those samples pickled in 15 per cent H_2SO_4 are most severely cracked in any given time and for a given treatment. The 5 and 30 per cent acids are roughly equal in the extent to which they cause cracking even though the 30 per cent acid has the highest hydrogen ion concentration. This, however, may be accounted for by the fact that the protecting sulphating action is greater.

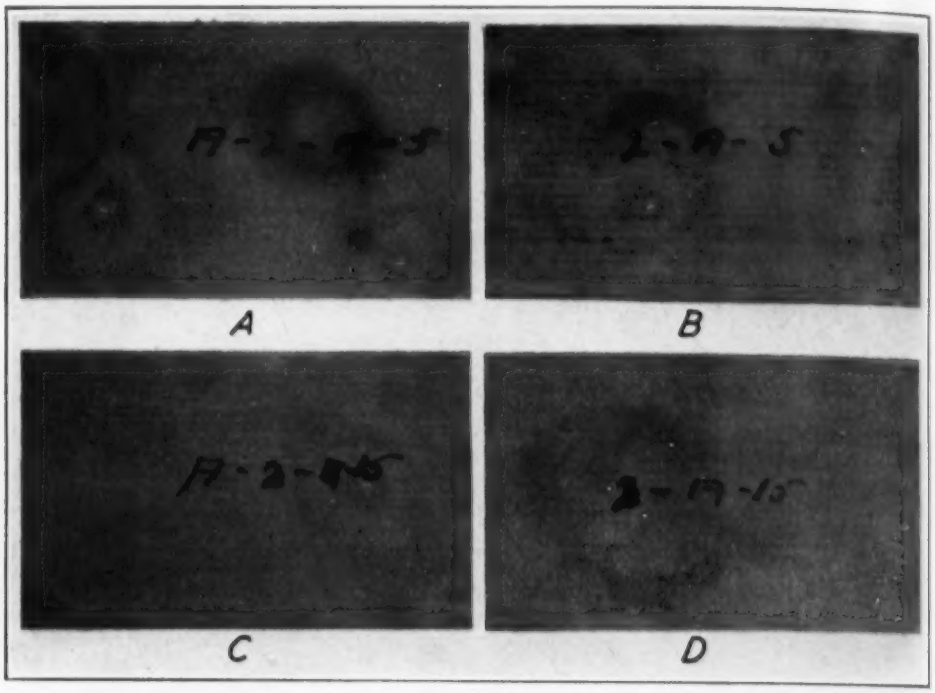


Fig. 6—Steel was Tempered at 300 Degrees Fahr. (150 Degrees Cent.) After Grinding and Before Pickling.
Specimens A and C Annealed at 1650 Degrees Fahr. (900 Degrees Cent.).
Specimens B and D Normalized at 1650 Degrees Fahr. (900 Degrees Cent.).
Specimens A and B Etched in 5 Per Cent Sulphuric Acid.
Specimens C and D Etched in 15 Per cent Sulphuric Acid.

Discussion of Results

The mechanism of stress generation during grinding is an interesting speculation, but, that stresses are generated, cannot be denied. The removal of metal by the wheel causes a considerable heating of a small volume of steel immediately beneath and this may be sufficient to raise its temperature to the plastic range. The metal naturally expands, and, since the rigidity of the surrounding metal prevents lateral expansion, only vertical expansion takes place causing a bulge which is at once leveled by the wheel. The remainder of the heated volume, however, is permanently upset and, as the wheel passes on, the hot metal is chilled rapidly. It now tends to shrink and, because there is actually less metal to fill the volume than is required, the metal is stretched and remains highly stressed. if not actually broken. The cracks, either those which appear at once, or those appearing after pickling, always seem to be at right angles to the direction of grinding.

This action may account also for the wavy appearance of some ground surfaces.

The presence of foreign inclusions and cementite network in steel being ground is very apt to cause excessive local heating resulting in greater stresses than are normally found. That, together with the fact that these are points of weakness, can logically account for the greater amount of cracking noticed on steels of this type.

The stresses introduced by grinding are considerably reduced by tempering previous to grinding and, if the tempering has reduced the hardness to Rockwell C 59-60, is difficult to introduce by grinding, additional stresses which are dangerous. It is also found that tempering after grinding is of considerable importance, in that it reduces the total stress to a safe point. This is especially true of precision parts which must hold their dimensions within very small limits, or of parts which must withstand high unit working stresses.

The effect of high quenching temperatures is more or less expected. Whether the actual grinding stresses are greater is not known, but when superimposed on higher quenching stresses the result is a greater total stress and more danger of cracking.

It is realized that hardened and ground steel is seldom, if ever, pickled, as was done in these experiments, but it must be understood that the pickling was but a method used to reveal stresses and, it is believed, to show differences of stress. However, hardened and unground steels are frequently pickled for scale removal and this may or may not be dangerous, depending upon the individual quenching stresses and their distribution. It is believed that if the part has been sufficiently tempered, there is no permanent damage resulting from pickling. Numerous experiments have shown that aging or slight reheating is quite sufficient to eliminate any temporary embrittlement due to absorbed hydrogen.

By pickling in increasing concentrations of either HCl or H_2SO_4, the results indicate that stresses of decreasing magnitude are picked out, and that pickling in a given concentration of acid for increasing length of time will also pick out lower and lower stresses. Mahin quotes Edwards as stating that the diffusion of atomic hydrogen through iron during pickling in H_2SO_4 increases up to 15 per cent and decreases beyond. This would account for the increased action of 15 per cent H_2SO_4 observed in these experiments.

There have been several ideas advanced as to the cause of cracking during pickling and with these there is no disagreement.

It is desired to suggest that, in addition to stresses developed by absorbed hydrogen, there is a considerable selective solution due to stress gradients and slight structural differences resulting from the heating during grinding. This selective solution would cause notching which would result in further stress concentrations and ultimate cracking.

Reheating, or tempering, releases and redistributes these stresses so that notching does not occur. As evidence of this, consider the spring experiments of Fuller.[4] Here after cold forming springs, they are copper plated and then tested. A loss of strength of more than 50 per cent is noted. But, upon heating a cold-formed spring in molten tin at 500 to 570 degrees Fahr. (260 to 300 degrees Cent.) and then plating as before, practically full strength is found. The tin coat was thought to protect the spring from hydrogen penetration, but it is also quite possible that sufficient stress had been so released to prevent permanent damage.

Conclusions

The results of these experiments seem to justify the following conclusions:

1. All grinding, whether done with correct or incorrect grinding practice, introduces stresses in the surfaces ground.
2. These stresses superimposed upon the quenching stresses may be sufficient to crack the steel.
3. The stresses introduced by grinding occur as gradients and that these are more dangerous than uniform stresses when subjected to the action of acid.
4. Uniformly quenched steel does not crack when pickled even though it is untempered.
5. Nitric acid does not produce cracking when stressed steel is immersed in it.
6. A weak solution of either hydrochloric or sulphuric acid will reveal high stresses and stronger solutions will reveal lower stresses in any given time of immersion.
7. Low stresses require a long time, and higher stresses a relatively shorter time to cause fracture in a given strength of acid.
8. 15 per cent sulphuric acid is the most effective of any of the acids tried in revealing stresses.
9. Low temperature heating before grinding decreases the intensity of stresses introduced during quenching.

10. Low temperature heating after grinding decreases the intensity of the stresses introduced by grinding.

11. Precision parts which are required to hold their dimensions to close limits should always be reheated to at least 300 degrees Fahr. (150 degrees Cent.) after grinding. Preferably this should be done after rough grinding and followed by careful finish grinding.

DISCUSSION

Written Discussion: By Chauncey E. Sweetser, research laboratories, Norton Company, Worcester, Mass.

The writer has found Mr. Brophy's paper extremely interesting and believes that the information presented has a high practical value. His studies in connection with the effects of quality of steel, heat treatment, and acid treatment should add much to our knowledge concerning the causes and prevention of cracks in hardened and ground steel.

This discussion of the subject paper is based on information gained in the study of a recurrent industrial problem that is frequently the cause of controversy between steel makers or heat treaters and grinders. When cracks appear in hardened and ground steels, which is the predominating cause, heat treatment or grinding? Conclusions, illustrative results and details concerning this study were reported in the May, 1930, issue of the Norton Company's publication, *Grits and Grinds*. Type of steel and methods of heat treatment and grinding were variables investigated. High speed steel and two alloy die steels were used in the experiments.

In each instance of cracking, the major cause must be determined by a study of the conditions and evidences involved. When little is known concerning the methods of grinding and heat treatment, the recommended procedure is to acid treat the sample in question and to study the cracks thus revealed with respect to their number, size, form and location. The study indicated that distinctly abusive grinding is required to crack steel that has received correct heat treatment as prescribed by steel makers. Such cracks were shown to occur in the form of a fine surface network. Only slight abuse by grinding is required to crack steel which has been heat treated in a manner to produce a high degree of strain or brittleness. These cracks have been found to be comparatively wide and deep, and frequently extend entirely through the section ground. Between these two extremes there is a range of possible combinations of heat treatment and grinding abuse that may cause cracking.

It is interesting to note that the results and illustrations presented by Mr. Brophy to show the influence of heat treatment agree in general with those obtained by the writer of this discussion.

Mr. Brophy has added a third factor to the problem, that of quality of steel, and has shown how cracking may be the result of a high dirt content. His illustrations of such cracks do not show their depth but indicate that in sur-

face form they are similar to those already described here as being typical of improper heat treatment.

The following comment might naturally be expected from one who is primarily interested in grinding. By intentionally omitting details and discussion of grinding methods, it would seem that Mr. Brophy has unintentionally placed exponents of this art on the defensive. It is agreed that all grinding introduces stresses in the surface ground. It should be emphasized, however, that the degree of such stresses depends on the conditions of grinding employed. A sensitive piece of steel can be finished successfully with proper grinding methods, whereas improper or abusive grinding would have ruined it.

Abusive grinding is defined here as grinding in which excessive heat is generated by heavy feeds or by the action of a glazed or dull wheel. Glazing may result from the use of a wheel that is too hard or from the use of machine conditions which cause the wheel to act hard. Noticeable surface discoloration is a danger signal, indicating the approach of, or an actual condition of, abusive grinding.

Written Discussion: By R. E. W. Harrison, chief engineer, Cincinnati Grinders, Inc., Cincinnati, Ohio.

Mr. Brophy's paper draws attention to an important phase in the fabrication of heat treated steel components. However, from the appearance of the slides presented at the meeting and the illustrations in Mr. Brophy's paper, it would appear that the work has been ground with a wheel which has resulted in minute surface burning of the work.

It is almost invariably possible to produce surface cracks in heat treated steel by using too hard and too fine a grinding wheel, or by using the correct grade of grinding wheel in such a way that a deep cut is taken into the work and a large amount of local heat generated.

The writer's experience indicates that while correct grinding practice will sometimes reveal flaws or cracks in heat treated steel, these cracks having existed prior to the starting of the grinding operation, the grinding operation itself can quite frequently be so conducted that it adds minute stresses and strains to a structure which is already internally stressed by the quenching operation following on the heat treatment. These internal stresses existing prior to grinding will occasionally reach a point where they are from 50 to 90 per cent of the strength of the steel, and it will be readily understood that a grinding operation so conducted that it adds stress to the already overburdened structure, may easily cause fractures to appear on the surface.

Grinding, no matter how conducted, invariably creates local heat, and this local heat naturally results in rapid expansion and contraction, which in turn create high surface stresses.

The writer believes Mr. Brophy is correct in his recommendation regarding a low temperature tempering or normalizing operation prior to and in between the various phases of the grinding operation, especially on those classes of work which call for a high degree of hardness and reliability in the finished product.

Experience in the manufacture of precision gages indicates that a repeated heating and cooling at relatively low temperatures quite appreciably changes the

size of the gages, grinding being an operation which generates local heat, it is, therefore, highly desirable that the piece be subsequently heated all over so that the stresses generated by the local heating of grinding be eliminated. The operation might well be called an accelerated seasoning process.

It is possible that Mr. Brophy in preparing his specimens, deliberately generated surface tensions and compressions by using too hard and fine a grinding wheel at too high a pressure.

The writer's contribution to this discussion is based on the thought that an impression might be created by Mr. Brophy's paper that grinding is primarily responsible for surface cracks, whereas experience indicates that although grinding will bring to the surface cracks which would otherwise not be revealed, the best safeguard is to release the quenching strains prior to rough grinding, and then to again release strains caused by the rough grinding by low temperature heating prior to finish grinding.

Even after taking the precautions mentioned above, it should always be borne in mind that it is necessary, when grinding steel components of extreme hardness, a very soft grade of grinding wheel should invariably be used, even though this may appear to make the ratio of wheel wear to metal removed excessive.

It has been proved time and time again that it is more economical in the long run to pay for a little more abrasive than to possibly spoil a number of highly valuable pieces of work during the final machining operation.

Mr. Brophy is to be congratulated on the presentation of an extremely interesting and useful paper.

H. H. Ashdown[1]: It would be interesting to learn if microscopical examination was made of any pieces after grinding and before acid immersion. Although there is no question of the subsurface being highly stressed I suggest many of the assumed stress lines which subsequently developed into cracks, were already incipient cracks.

Some time ago I had occasion to examine a ring which had been hardened and ground and shortly after grinding was dropped onto a metal floor plate and broke into several pieces and although there were only two or three isolated visible surface cracks,—under the microscope the subsurface for some depth revealed a number of very fine cracks. These cracks were subsequently confirmed by boiling the sections in a chloride solution. The sections were thoroughly washed and dried and a few hours later fine rust lines appeared and developed corresponding with the cracks. If these cracks are proved I suggest even at room temperature the atomic hydrogen may slowly resolve into the molecular form creating sufficient pressure to develop the cracks. The effect of the light annealing before acid immersion may only retard the cracks developing.

With regard to the suggestion of atomic hydrogen reducing any iron oxide present forming water and initiating pressure may be deserving of more careful thought. If water was formed as suggested,—in pickling commercial steel plates, which steel unquestionably contains both free and dissolved oxides, surely

[1]Consulting metallurgist and engineer, Y. M. C. A., South Chicago.

all the hydrogen generated should quickly react with the oxides forming water.

Here is an exhibit of commercial steel plate which was pickled in H_2SO_4 and subsequently galvanized, and while in the galvanizing bath developed a fine crop of blisters, some as large as hen's eggs. These were drilled under water and the gas liberated from considerable pressure was collected and examined and proved to be practically pure hydrogen.

These blisters were originally unwelded blowholes and I suggest during pickling the atomic hydrogen in its progress through the plate was arrested at these discontinuities and on heating in the galvanizing bath resolved into the molecular form developing a very great pressure.

These plates were about $\frac{1}{16}$" thick and this was no isolated case. I submit this only to illustrate the great pressure effect of the hydrogen and also to question its reaction with the oxide in the steel.

A. H. d'ARCAMBAL[2]: It has been stated at times that more tools are lost in the grinding operation than during hardening. While I believe this statement to be somewhat exaggerated, it is true that thousands of tools are lost monthly due to poor grinding. Mr. Brophy has brought out several points in his paper which support tests conducted in our plant. For example, it is possible to take taps, purposely overheat them in hardening, then check the same during grinding, even though the grinding practice is satisfactory. Taps properly treated will not develop cracks when correctly ground. Therefore the grinding department should not always be blamed when grinding cracks develop. Quite often tools in service are improperly ground, using too hard a wheel, or a loaded wheel. The very fine cracks developed usually are not revealed until the tools are placed in service. High speed tools will in the majority of cases crack during the grinding operation if they are not properly tempered, so it is always very important to see that the tools are properly quenched and tempered so as to give the operator doing the grinding operation an even break.

Hardening room foremen as well as grinding room supervisors should carefully read this excellent paper by Mr. Brophy as there are several important points brought out in the same.

Author's Closure

It is apparent from Mr. Harrison's discussion, and also that of Mr. Sweetser, that the paper places the grinder on the defensive. I wish to emphasize again that this was not my intention, but rather to place some of the responsibility for grinding cracks on the heat treatment.

It must be realized that when two conditions may be varied, in order to study the influence of either, the other must be kept constant. The grinding practice used in this investigation, while perhaps not ideal, was kept the same for all heat treatments, so that differences in cracking tendencies are due to the difference in heat treatments.

Regarding the occurrence of incipient cracks in the steel before grinding, it is to be remembered that each piece was pickled both in nitric and sulphuric

[2]Consulting metallurgist, Pratt and Whitney Co., Hartford, Conn.

acids previous to grinding to reveal such cracks. Only in the case of high temperature quenching were any found.

The occurrence of cracks after grinding is not necessarily dangerous because these may be seen upon careful visual examination and the parts scrapped. The important point is, however, that the absence of cracks does not indicate the absence of dangerous stresses, which may later develop into cracks and cause failure of the part. This condition may be at least partly corrected by proper heat treatment.

In answer to Mr. Ashdown's discussion, the incipient cracks which he speaks of were not present in all the steels before pickling as was shown by immersion in nitric acid immediately after grinding. In a good many of the samples after grinding they were not developed until pickled in sulphuric or hydrochloric acids for a certain length of time, depending upon the degree of stress and the concentration of acid. As to the theories of acid embrittlement, Mr. Ashdown's idea conforms with the usually accepted idea of hydrogen embrittlement of steel. This is the theory which appeals to me most; that the atomic hydrogen arrives at a discontinuity and forms molecular hydrogen thus building up a pressure sufficient to crack the steel or leave it highly stressed.

OCCURRENCE OF FISH-SCALE IN HIGH SPEED STEEL

By G. R. Brophy, R. H. Harrington and A. W. Merrick

Abstract

Fish-scale in high speed steel is the result of abnormal grain growth in the austenite due to critical strains. The strains are introduced either thermally or mechanically.

Thermal strain: Introduced by air cooling from 850 degrees Cent. (1560 degrees Fahr.) for the 14 per cent tungsten steel and 1150 degrees Cent. (2100 degrees Fahr.) for the 18 per cent steel.

Mechanical strain: Introduced by deformation of the steels in their austenitic ranges.

Introduction

BY its nature and the manner of its occurrence, fish-scale has an interesting history that is of considerable importance to the success of high speed steel. Repeated hardening without intermediate annealing seems to be the only recognized cause. But, like a will-o'-the-wisp, it has occurred here and there seemingly under varying conditions.

Although fish-scale undoubtedly is as old as high speed steel, its recognition as a distinctive structure has been comparatively recent. In a paper on "Lathe Breakdown Tests"[1], published in 1922, the abnormal grain size in high speed steel is referred to as fish-scale in the statement: Tools that failed "often show fish-scale fractures and in cutting tests, generally poor and erratic results are obtained." In a publication of the same year on "Dimensional Changes of High Speed Steel in Heat Treatment"[2] is written: "Hardening of lower tungsten steels often gives coarse grain and brittleness. Higher tungsten steels give finer grained structure."

[1]H. J. French and J. Strauss, Transactions, American Society for Steel Treating, 1922, p. 1125.

[2]M. A. Grossmann, Transactions, American Society for Steel Treating, 1922, p. 695.

A paper presented before the Twelfth Annual Convention of the society held at the Stevens Hotel, Chicago, September 22 to 26, 1930. The authors are members of the society and are associated with the research laboratories, General Electric Co., Schenectady, N. Y. Manuscript received July 25, 1930.

In a paper published in 1923[3], French, Strauss, and Digges describe the production of "flaky" steel or "fish-scale" by reharden-ing a tool, part of which had been heated to annealing temperatures. They established the fact that, once produced, fish-scale can be completely removed only by combined heavy forging and long annealing treatments. Thus it is quite apparent that the prevention is worth many times the cure.

A review of the whole of the literature on high speed steel would be unwieldy and unnecessary to the purposes of this paper. The papers referred to specifically seem to contain the available data bearing on the occurrence and nature of fish-scale.

Occurrence of Fish-Scale Through Thermal Treatment

It was noticed by one of the authors that when a 14 per cent tungsten steel was heated above some minimum temperature, air-cooled, and then hardened that the fracture was scaled and also that an 18 per cent tungsten steel did not respond to the same treatment. If slowly cooled however, in the furnace from the same temperature and then hardened, the normal silky fracture was obtained.

A bar of 14 per cent steel was heated to 1275 degrees Cent. (2330 degrees Fahr.) at one end, the other end being kept cold. After air cooling the whole bar was heated uniformly to 1275 degrees Cent. (2330 degrees Fahr.) and oil quenched. Upon fracturing at several points the fish-scale structure appeared on the end which had been heated but disappeared suddenly at the point corresponding to a temperature of 800-900 degrees Cent. (1470-1650 degrees Fahr.) on the temperature gradient of the prequench heating. This indicated a critical temperature above which the steel must be heated to put it in a condition to develop fish-scale.

A. Following this lead, steels were obtained of three general chemical compositions and two mechanical treatments as follows:

1. 14 per cent tungsten, 1 per cent vanadium and hammered.
2. 14 per cent tungsten, 2 per cent vanadium and hammered.
3. 14 per cent tungsten, 2 per cent vanadium and rolled.
4. 18 per cent tungsten, 1 per cent vanadium and hammered.
5. 18 per cent tungsten, 1 per cent vanadium and rolled.

[3] "Effect of Heat Treatment on Lathe Tool Performance" by H. J. French and J. Strauss, J. V. Digges. TRANSACTIONS, American Society for Steel Treating, 1923, p. 353.

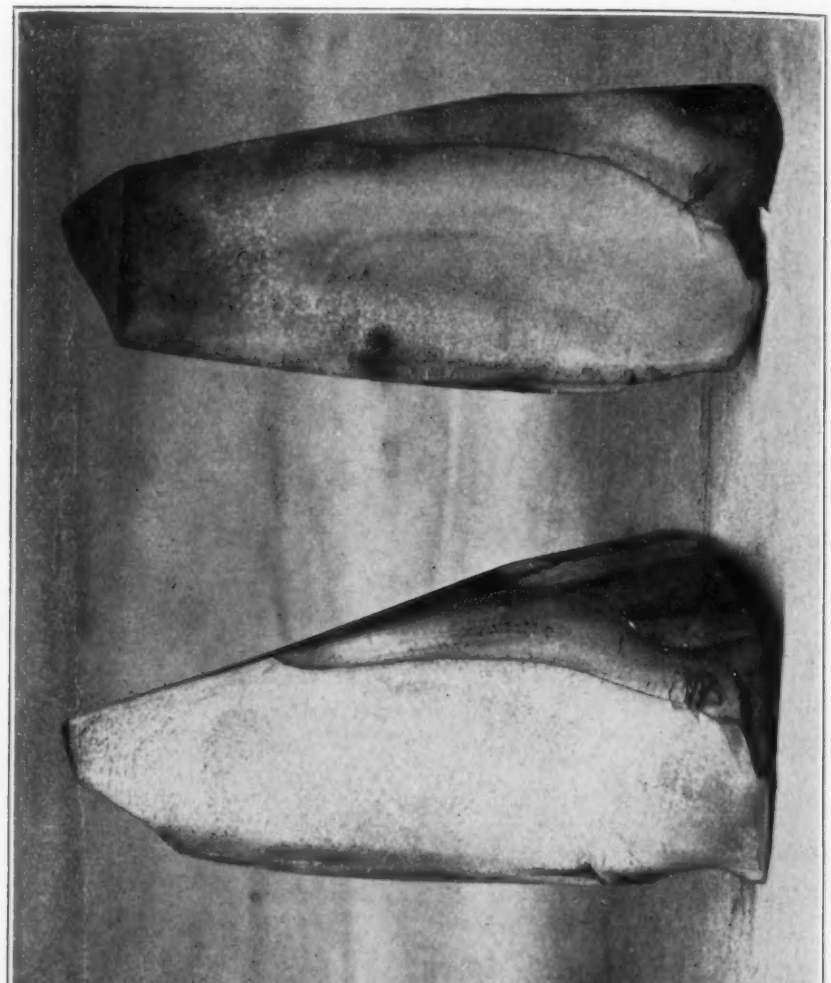


Fig. 1—Fish-Scale in High Speed Steel Showing Tools that Have Failed.

A series of about 24 samples, 1½" x ⅜" x ⅜", and notched for fracturing, was made from each steel. The following heat treatments were used:

(a) Although all of the steel had been annealed after forging and rolling, the five series were reannealed at 850 degrees Cent. (1560 degrees Fahr.) to eliminate effects of notching.

(b) One sample from each series was air-cooled from each temperature between 100 degrees Cent. and 1200 degrees Cent. (2190 degrees Fahr.) increasing by 50-degree increments.

(c) Each series was then given the regular hardening treatment consisting of (1) preheating to 800-850 degrees Cent. (1470-1560 degrees Fahr.) (2) oil quenching from 1275 degrees Cent. (2330 degree Fahr.) followed by (3) a temper to 600 degrees Cent. (1110 degrees Fahr.).

Each sample was then fractured and examined for fish-scale. Figs. 2 to 6 show the results.

Fig. 2 shows the series made from the hammered 14 per cent tungsten and 1 per cent vanadium. A trace of fish-scale was observed in the 700 degrees Cent. (1290 degrees Fahr.) sample. Check samples were air cooled from 675 and 725 degrees Cent. (1250 and 1340 degrees Fahr.), hardened, and fractured. These showed no fish-scale. Thus the 700-degree Cent. sample appears to be but a vagrant. All of the samples from 850 to 1200 degrees Cent. (1560 to 2190 degrees Fahr.) showed beautiful fish-scale fractures. A check sample, air-cooled from 825 degrees Cent. (1515 degrees Fahr.) previous to hardening showed a partial fish-scale fracture.

Fig. 3 shows the series made from the hammered 14 per cent tungsten and 2 per cent vanadium. The 300-degree Cent. (570-degree Fahr.) sample proved a vagrant, showing a small spot of flake. However, all of the samples from 850 to 1200 degrees Cent. showed 100 per cent fish-scale sections.

Fig. 4 shows the series made from the rolled 14 per cent tungsten and 2 per cent vanadium steel. In some of the samples air-cooled from temperatures below 800 degrees Cent. (1470 degrees Fahr.) there is a tendency for a core-and-frame structure of fish-scale. As in the other 14 per cent tungsten steels, the samples from 850 to 1200 degrees Cent. show 100 per cent fish-scale.

Fig. 5 shows a similar series made from hammered 18 per cent

Fig. 2—Fish-Scale in 14 Per cent Tungsten, 1 Per Cent Vanadium High Speed Steel, Hammered.

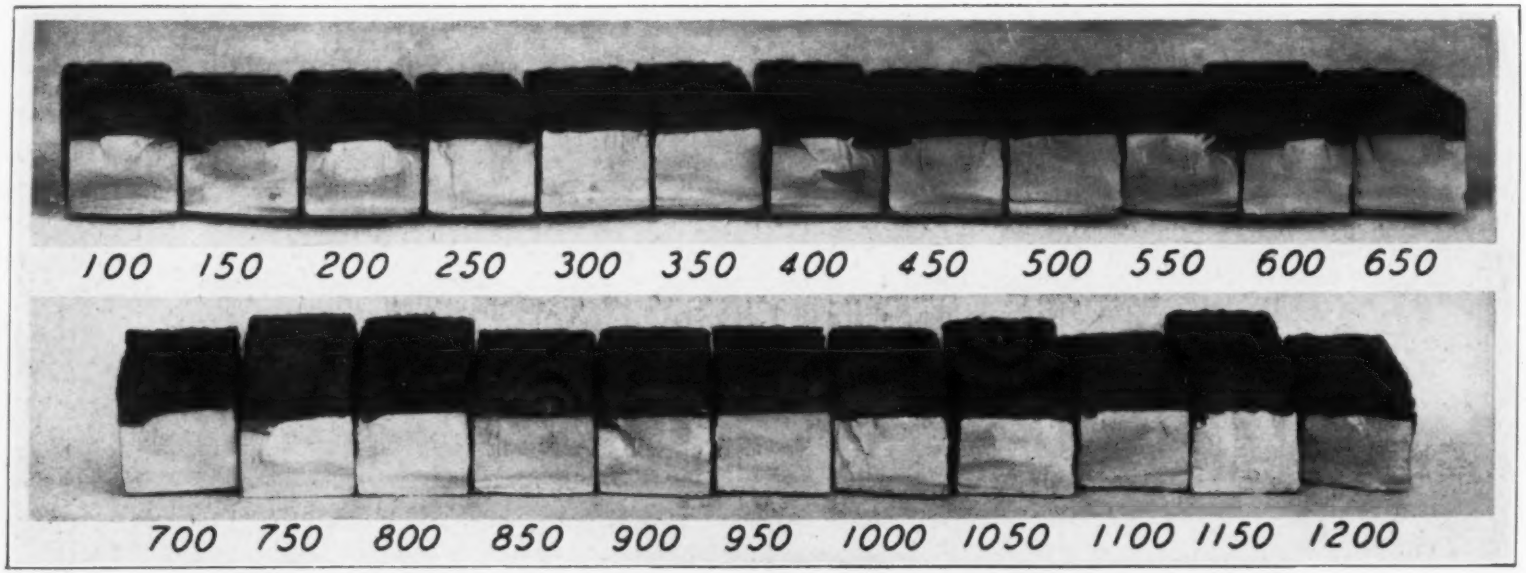


Fig. 3—Fish-Scale in 14 Per Cent Tungsten, 2 Per Cent Vanadium High Speed Steel, Hammered.

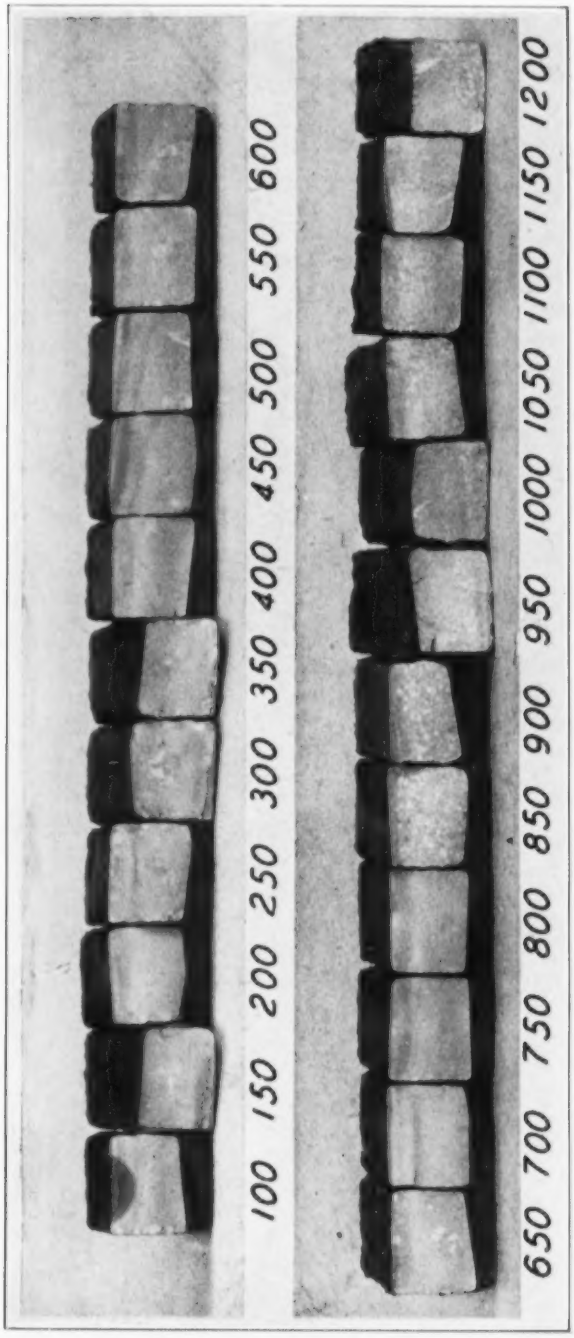


Fig. 4—Fish-Scale in 14 Per Cent Tungsten, 2 Per Cent Vanadium High Speed Steel, Rolled.

tungsten and 1 per cent vanadium steel. The 1150-degree Cent. (2100-degree Fahr.) sample showed traces of fish-scale and the 1200-degree Cent. (2190-degree Fahr.) sample showed 100 per cent fish-scale.

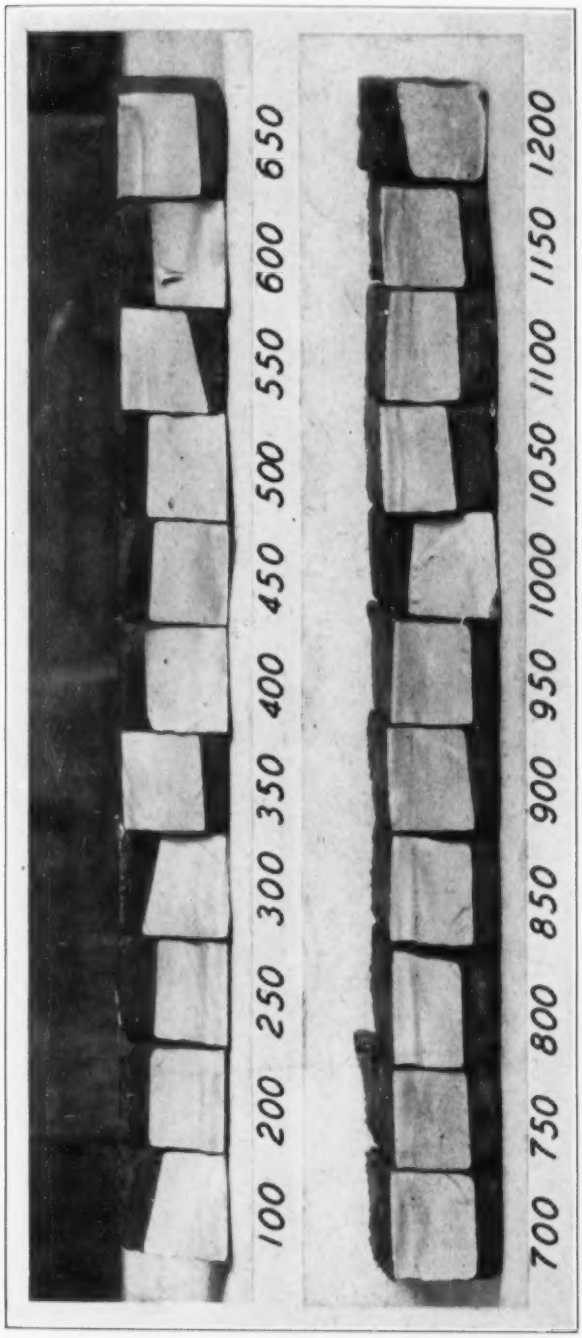


Fig. 5—Fish-Scale in 18 Per Cent Tungsten, 1 Per Cent Vanadium High Speed Steel, Hammered.

Fig. 6 shows the series made from the rolled 18 per cent tungsten steel and only the 1250-degree Cent. (2280-degree Fahr.) sample showed 100 per cent fish-scale.

It appears therefore that whether the steel is rolled or ham-

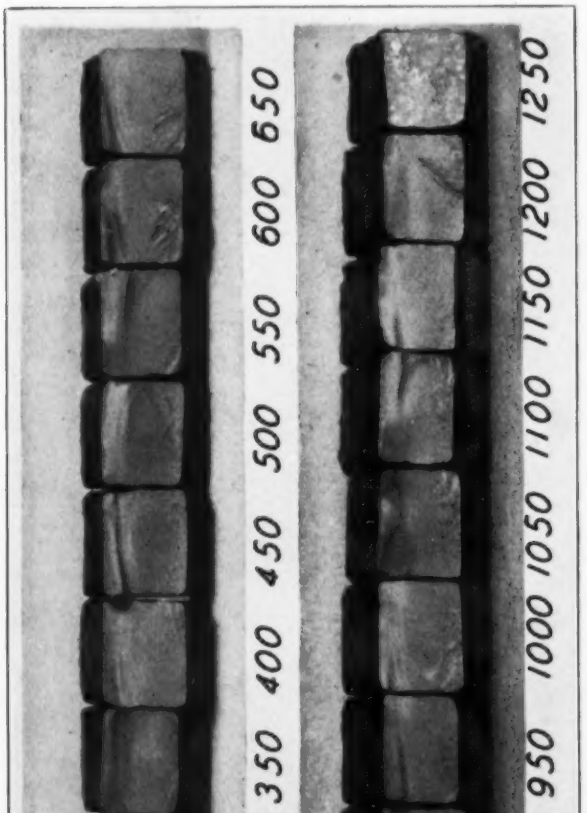


Fig. 6—Fish-Scale in 18 Per Cent Tungsten, 1 Per Cent Vanadium High Speed Steel, Rolled.

mered is of little importance but that its composition is the major factor.

B. Endeavoring to find at just what point in the heat treat-

ment cycle fish-scale occurred, four samples of the 14 per cent tungsten, 1 per cent vanadium steel were heat treated and fractured as follows:

1. The first sample, in the annealed condition, was fractured as annealed, and, of course, showed no fish-scale.

2. The second annealed sample was heated to 900 degrees Cent. (1650 degrees Fahr.) and air-cooled. This treatment is known to produce fish-scale after hardening treatment. The air-cooled sample was fractured and no fish-scale was apparent.

3. Sample No. 3 was cooled from 900 degrees Cent. (1650 degrees Fahr.), as was sample No. 2, heated to 1275 degrees Cent. (2330 degrees Fahr.) and quenched in oil. This sample showed 100 per cent fish-scale.

4. Sample No. 4 was treated similarly to No. 3, but was tempered at 600 degrees Cent. (1110 degrees Fahr.) after quenching. When fractured it appeared to have fish-scale identical to that in sample No. 3. Thus it is shown that fish-scale develops in the steel during the hardening heat and persists, unchanged in the tempered condition.

These results indicate at this point that strain set up probably by solution during the prequench treatment is responsible for abnormal grain growth at the hardening temperature.

C. In order to set up different degrees of strain and study the results, the following experiments were tried:

1. The first sample of annealed 14 per cent tungsten and 1 per cent vanadium steel was heated to 900 degrees Cent. (1650 degrees Fahr.), held one hour and transferred without cooling to a furnace at 1275 degrees Cent. (2330 degrees Fahr.) from which it was quenched in oil and followed by a 600 degrees Cent. tempering treatment. When fractured it showed no fish-scale.

2. The second sample was reannealed from 900 degrees Cent. and hardened and tempered as in regular practice. When fractured this sample was free from flakes.

3. The third sample of this annealed steel was air-cooled from 900 degrees Cent., then hardened and tempered. Upon fracturing this sample gave a beautfiul coarse-grained fish-scale.

4. The next sample was oil-quenched from 900 degrees Cent. previous to quenching and tempering as before. Upon fracturing, this sample was 100 per cent fish-scale, but of finer grain size than No. 3.

5. The fifth sample was water-quenched from 900 degrees Cent. (1650 degrees Fahr.) previous to hardening and tempering. Upon fracturing, it showed a fish-scale fracture that was of finer grain size than that of sample No. 4.

Thus it is seen that maximum grain growth occurs under the strain accompanying air cooling rates from temperatures above 850

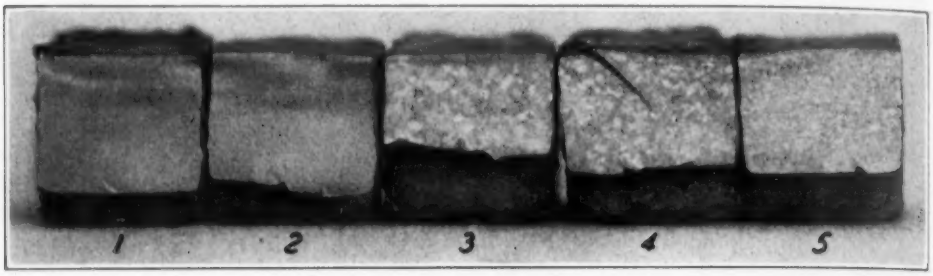


Fig. 7—Effect of Different Rates of Cooling in the Prehardening Treatment on the Production of Fish-Scale in 14 Per Cent Tungsten, 1 Per Cent Vanadium High Speed Steel.

No. 1—1 Hour at 900° C. Preheat—then Hardened.
No. 2—Annealed from 900° C. and then Hardened.
No. 3—Air-Cooled from 900° C. and then Hardened.
No. 4—Oil-Quenched from 900° C. and then Hardened.
No. 5—Water-Quenched from 900° C. and the Hardened.

degrees Cent. (1560 degrees Fahr.) in the 14 per cent tungsten steels and that without this cooling no fish-scale develops. The fractures of the steel in these five conditions are shown in Fig. 7.

Occurrence of Fish-Scale as Related to Mechanical Treatment

A ½-inch square bar of the 14 per cent tungsten and 1 per cent vanadium steel was heated to a temperature of 950 degrees Cent. (1740 degrees Fahr.), withdrawn from the furnace, hammered with a ball peen in several spots by hand, without allowing it to cool below 850 degrees Cent., and replaced in a furnace at 1275 degrees Cent., from which temperature it was quenched in oil, followed by a 600 degrees Cent. temper. This bar was fractured through the hammered sections and each section showed fish-scale in areas concentric with the depressions and at varying distances from the surface hammered and depending apparently on the amount of deformation. Fig. 8 shows one of these fractures. Fish-scale of coarse grain size forms a cup around the hammered spot with the maximum grain size farthest from the impression. It is then

apparent that a slight strain is the optimum condition for grain growth. It is interesting to note in Fig. 7 that the sample with only slight strain resulting from air-cooling from 900 degrees Cent. (1650 degrees Fahr.) gave a coarser grain size than the more severe strain caused by oil and water quenching from the same temperature.

A section through one of the hammered areas was polished, etched, and photographed at about 30 diameters. This is shown

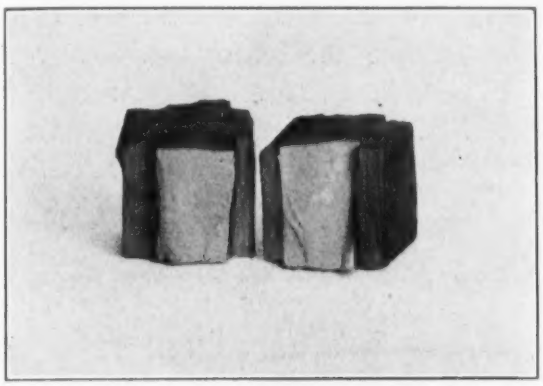


Fig. 8—See Text on Page 450 for Description of these Specimens.

in Fig. 9. The grain size of the austenite is greatest at the border of the volume affected by the hammering and therefore where the strain was slight.

The 18 per cent tungsten, 1 per cent vanadium steel when hammered under the same temperature conditions also produces fish-scale, but simple thermal treatment below 1150-1200 degrees Cent. (2100 to 2190 degrees Fahr.) does not produce strain enough to cause grain growth.

An annealed bar of each steel was machined with uniform thickness but a tapering width, and then placed in a tensile testing machine and pulled at room temperature until fractured. In this manner all degrees of strain were obtained. The bars were then oil-quenched from 1275 degrees Cent. (2330 degrees Fahr.) and fractured at several points, but no sign of fish-scale was found. This result proves that if the strained steel is in the alpha form no abnormal grain growth develops at the high temperature. This is to be expected since the steel passes through a phase change which would radically change the grain size and remove strain.

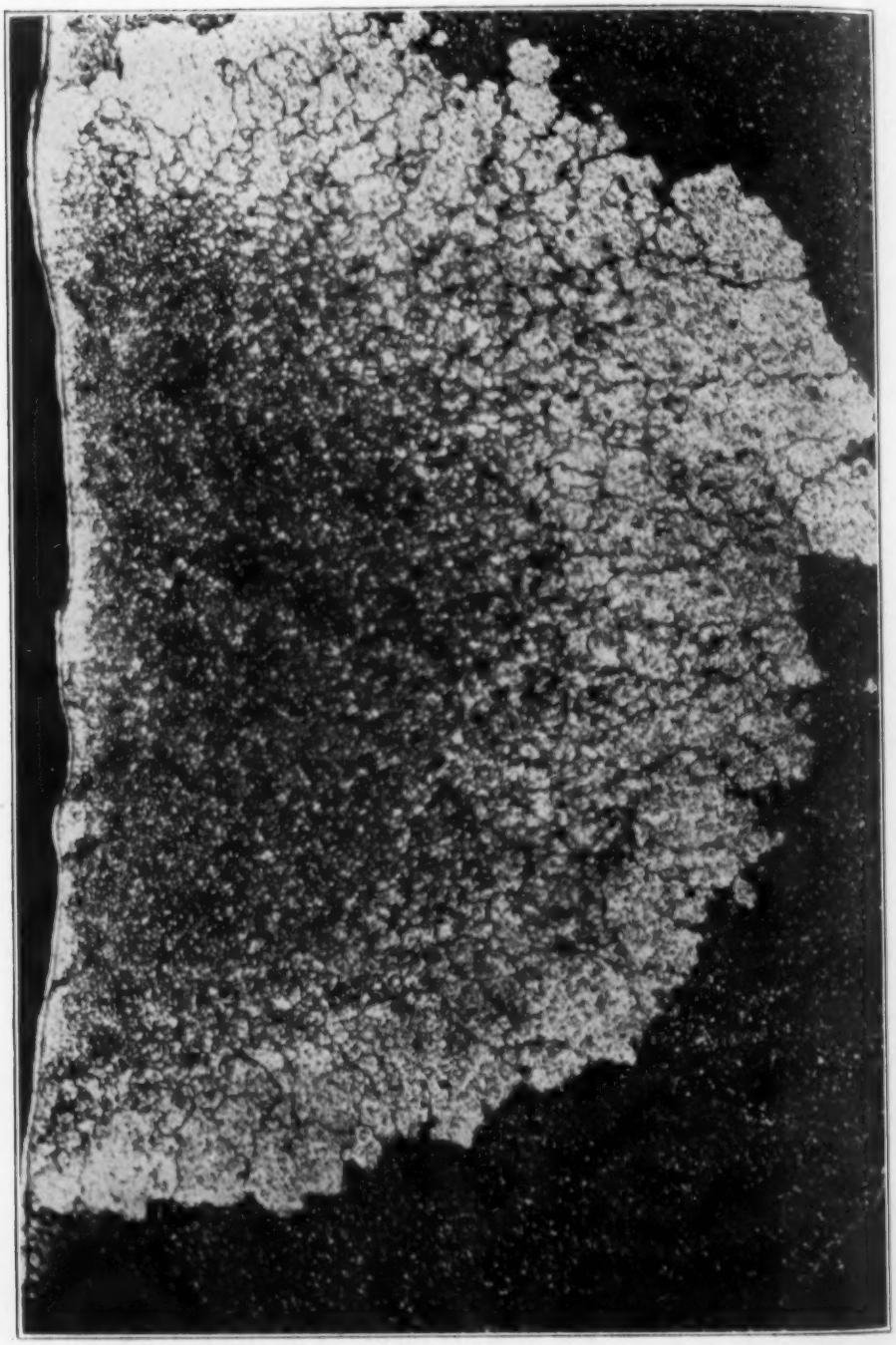


Fig. 9—Section through One of the Hammered Areas of Fig. 7. Magnified X 30.

Persistence of Fish-Scale

Samples of 14 per cent tungsten steel exhibiting fish-scale struc-

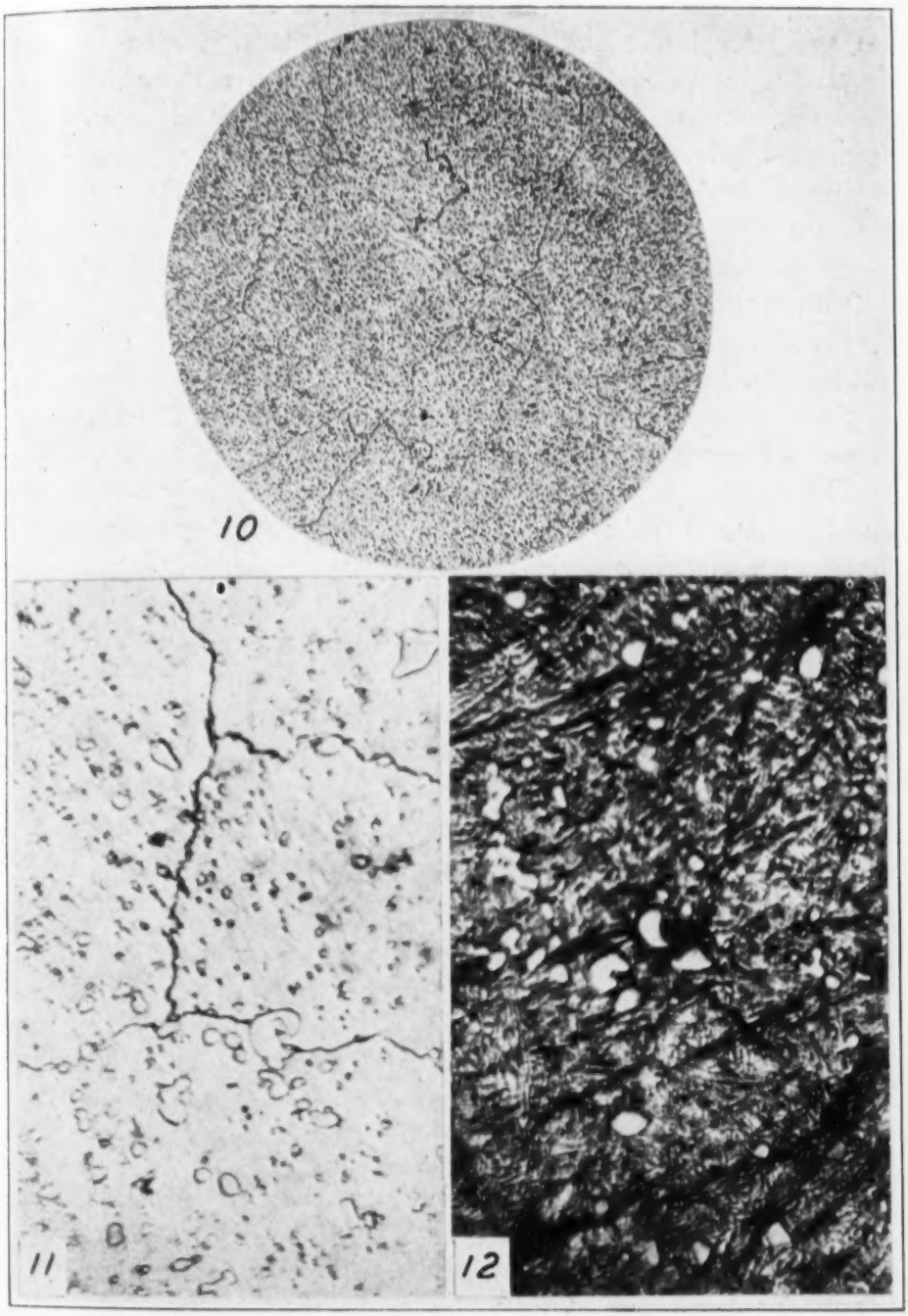


Fig. 10—Grain Size in the Usual Fish-Scaled 14 Per Cent Tungsten High Speed Steel ×100.
Fig. 11—Same Specimen at × 1500.
Fig. 12—Same Specimen as Fig. 10 after Tempering at 600° C. × 1500.

ture were annealed at 850 degrees Cent. (1560 degrees Fahr.) for two and four hours and cooled in the furnace. When fractured the sample annealed for two hours still showed a large grain size, while that annealed for four hours showed a considerably refined grain. However, upon rehardening at 1275 degrees Cent. (2330 degrees Fahr.) each again showed a 100 per cent fish-scale structure.

Other samples were heated to 850-900 degrees Cent. (1560 to 1650 degrees Fahr.) and air-cooled and then annealed for varying lengths of time at 850 degrees Cent. to relieve strain. These were then hardened and it was found that at least one hour at temperature for the sizes used was necessary to eliminate sufficient strain and to prevent fish-scale upon hardening.

Once produced, the critical strain is elminated only after drastic annealing and if by chance fish-scale is produced, heat treatment alone is not sufficient to eliminate it.

Photomicrographs of Fish-Scale

Fig. 10 shows at 100 diameters the grain size in the usual fish-scaled 14 per cent tungsten steel. This sample had received the usual preheat and hardening heat but was not tempered.

Fig. 11 shows the same sample at 1500 diameters. The structure is seen to be austenitic with the characteristic carbide distribution. This photomicrograph shows a normal grain surrounded by three large abnormal grains.

Fig. 12 at 1500 diameters shows the same steel after a 600-degree Cent. temper. The development of martensite obscures the grain boundaries and presents a normal structure.

Conclusions

The evidence presented here indicates that the fish-scale structure frequently found in hardened high speed steel is another example of abnormal grain growth resulting from more or less critical strain for the temperature used in hardening. The laws governing abnormal grain growth are well established and need no further elaboration.

Two sources of strain are recognized, thermal and mechanical. The effects of mechanical straining are identical with those reported

elsewhere in the literature for other metals but the introduction of thermal strain sufficient to cause abnormal grain growth is believed not to have been shown before. It is necessary, however, to have a considerable amount of austenite present in the steel at the time of straining and it is probable that it is the strained austenite which shows the greatest growth activity at the hardening temperatures. As a possible source of thermal strain we have considered the retention of carbides and tungstides in solution, rather than by the cooling stresses alone. This phase of the subject, however, is receiving further study.

It is shown that the 18 per cent tungsten steel requires a prequench treatment from a considerably higher temperature to produce sufficient strain to cause abnormal grain growth. While the threshold value of the strain may not be any different in the two steels, the presence of more tungstides in the 18 per cent tungsten steel undoubtedly act as grain growth inhibitors, as does pearlite and other impurities in low carbon steel, and in order to remove these inhibitors it is necessary to go to a higher temperature to cause their solution. Thus it is that 18 per cent tungsten steel is more resistant to the occurrence of fish-scale.

While it is well known that annealing will remove the strain it is here shown that annealing must be for extended periods of time to thoroughly remove all previous effects of solution, or mechanical straining. But, if once produced, then fish-scale cannot be removed by simple annealing, but will recur on hardening in spite of any annealing.

Frequently in rough grinding a tool to shape, local temperatures are reached sufficiently high to thermally strain the steel, so that care should be exercised in this operation; and, to be on the safe side, the tool should be thoroughly annealed previous to hardening.

It is probably needless to say, at this point, that tools such as large lathe tools should be annealed between successive nosing and hardening operations, but it is not always done.

If deformation of the steel occurred in its austenitic range and is followed immediately by hardening, the chances are excellent for grain growth locally. This probably accounts for the vagrant samples found in these experiments. To eliminate this possibility,

tools should be carefully handled while hot to prevent striking one against another or deforming in any way.

The previous work on abnormal grain growth has shown that heavier strains are critical for lower temperatures, so that there are good possibilties for fish-scale to be produced at temperatures lower than those used for hardening high speed steel. Therefore, the mill, in rolling or hammering bars, should so control the heating and cooling, and the amount of reduction and the reheating to insure against producing fish-scale, because, obviously if formed at this stage, no amount of care in handling the tools will eliminate this defect.

DISCUSSION

C. B. SADTLER:[1] I was very much interested in hearing this paper. The point that struck me most favorably was the proof that was given for the close relationship between what is seen in the fracture and what is seen in the photomicrographs. Personally, I have had several arguments on my hands as to that very identity. Long before I had any very good proof, my belief has been that there was that identity. My arguments have been mainly with producers of steel who are very reluctant, at least two with whom I have had recent discussion were, to admit that identity. I have satisfied myself, however, by sufficient microexaminations of as quenched but not drawn material, which, at least metallographically, is austenitic, although it is not austenitic in the sense of being nonmagnetic, but from a metallographic point of view it looks austenitic and it is usually called that. A large number of such observations have convinced me that this austenite does occur at times in large grains which, even at low magnifications of 100 diameters, still look plenty big. Now, as to the causes for the formation of these grains. That is something interesting to people who use high speed steel. The surest way, as has been pointed out, and the method best known, is by repeated rehardening without reannealing. I have rarely heard of a case where that failed to produce at least some fish-scale. I have felt that it might be produced by methods which the authors mentioned. They were fortunate in having more opportunity than I have had in trying out a number of conditions in regard to thermal strains and mechanical strains, on which distinction I am not quite clear. My efforts were confined to a few attempts such as cutting a tapered slab from a billet, and then forming the piece of billet as a whole in such a way that the thin portion would receive no working by the steam hammer, while the end which was the thicker when placed on the hammer anvil would receive considerable working. Upon cutting such a piece into small pieces and subjecting them to a variety of hardening conditions, considerably to my chagrin and disappointment I was not able to develop the fish-scale fracture which I had anticipated. After quite a few attempts, I was still unsuccessful. This led me to the tentative conclusion that the formation

[1]Metallurgist, Barber-Colman Co., Rockford, Ill.

of fish-scale fracture must be governed by conditions other than that of mere critical deformation, which was the principle upon which I was working.

While I have not had the opportunity to digest the information presented in the paper and discussion, I would like to venture the opinion that there are some contributory factors such as submicroscopic obstructing agents functioning in very much the way that thoria is known to function in the case of tungsten, where detailed work has so thoroughly demonstrated that various percentage of obstruction agents, rates of heating, and sojourn at various temperatures exert all important influences on germinative grain growth. I offer that suggestion with the hope that the authors of the paper may give such comment as they are willing on that phase of the subject.

A. H. d'ARCAMBAL:[2] Several years ago we used quite a tonnage of the low-tungsten high-vanadium type of steel described in Mr. Brophy's paper. We found that fish-scale was quite noticeable on samples not properly annealed before rehardening. At times we would try a rapid anneal after the hardening operation, should it be necessary to reharden the tool, and we found that unless we fully annealed the hardened tool, we were quite liable to obtain a fish-scale fracture. In regard to 18 per cent tungsten type of high speed steel, we find it impossible at times to obtain a fish-scale fracture, even on repeated hardenings without annealing.

A few years ago we were preparing a few specimens of 18 per cent tungsten high speed steel to illustrate fish-scale fracture, and were not successful in producing this structure, even though the specimens were rehardened several times without first annealing. As this objectionable structure does occur at times, however, all high speed steel tools should be pack annealed to a maximum Brinell of 241 before being rehardened.

SAM TOUR:[3] The authors have discussed mainly 14 per cent and 18 per cent tungsten steels. I would like to add a little bit with regard to "hot-working" die steels of the 8 to 10 per cent tungsten type and some of the newer die steels of the 5 per cent tungsten type. In those steels we find fish-scale structures quite often in the forgings as produced at the mill. To date we have found no method of heat treating to overcome that structure. Steels showing this structure are low in impact strength and tend to develop large cleavage when subjected to sudden temperature changes in service. It appears as though this structure in these two steels is not a function of amount of reduction in forging but connected with both the maximum and the finish forging temperatures.

J. V. EMMONS:[4] The authors are to be congratulated for their excellent treatment of this subject. The so called fish-scale fractures of high speed steel have been long known in the realm of practical work, but there is too little information of scientific value on the subject.

It might be mentioned that there is another name for this type of fracture which has a certain amount of usage. It is sometimes called a "marble fracture."

[2]Consulting metallurgist, Pratt and Whitney Co., Hartford, Conn.

[3]Vice president, Lucius Pitkin, Inc., New York City.

[4]Metallurgist, Cleveland Twist Drill Co., Cleveland.

Some high speed steels, particularly the high tungsten types, are very resistant to the formation of this type of fracture. Cases have been observed where three or four repeated hardenings at high temperatures, only developed traces of this condition.

The best methods of preventing this fracture are, careful regulation of forging and rolling temperatures together with thorough annealing between heating operations.

Many cases have been observed where tools showing the fish-scale or marble fracture, have had excellent cutting quality and did not appear to have been damaged by its presence.

It has not been found possible to refine this fracture, when once found by heat treatment alone. It is, however, possible to entirely dispel the marble appearance by means of hot work.

Authors' Closure

The first speaker questioned the distinction between thermal and mechanical strain. There really is no distinction between them. They are both mechanical strains. One is caused by hammering and deformation of the steel and the other is caused by simple thermal stresses or, perhaps, the solution of carbides or tungstides which strain the austenitic lattice.

The question of inhibitors to grain growth is interesting. As we pointed out, the 18 per cent tungsten steel does not form the so called fish-scale structure—(or we might say the marble structure which undoubtedly is a better name)—because of the sluggishness of the tungstides, which was brought out by the fact that it was necessary to go to higher temperatures or longer time to produce the necessary straining.

NITRIDING IN PACKING MATERIALS AND AMMONIA

By A. B. Kinzel and John J. Egan

Abstract

An improved method of nitriding is herein described, which consists essentially of packing the work to be nitrided in suitable finely divided materials. These are inert to the reaction and may be nonmetallic or metallic. Magnesia, copper and prenitrided nitriding steels are very effective. The nitriding is carried out as usual in ammonia but less time is required for a given depth of case. Corrosion resistant cases have been produced by these means in four hours. The packing materials also act to give uniform nitriding, making the process independent of the condition of the container and circulation of the ammonia. The process is applicable to both chromium-aluminum and chromium-vanadium nitriding steels and should find ready commercial application.

Introduction

THE advantages of nitrided cases are many, the most notable being their hardness, corrosion resistance and freedom from distortion in application. Their use is rapidly increasing, and problems connected with the nitriding process have become more and more important as industrial applications have developed. Of the problems facing the industry today, not the least important is the matter of curtailing the time of the nitriding cycle. Equally important is the ability to reproduce cases in a satisfactory manner, not only from heat to heat but also on a number of specimens in a large container in a single heat. Much effort has been spent in connection with the solution of these problems, and at the present state of the art the controlled ammonia dissociation, the proper circulation of the nitriding gases and the variation of temperature cycles generally result in commercially satisfactory nitriding. In the experimental work herein described the authors have succeeded in obtaining a simple, direct, and satisfactory solution of both problems. This has been effected by the use of suitable packing materials.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The authors, who are members of the society, are associated with the Union Carbide and Carbon Research Laboratories, Long Island City, N. Y. Manuscript received June 7, 1930.

Evaluation of the Case

Before attempting a study of the various factors involved in nitriding, it is essential that a satisfactory method of evaluating a given case be established. The most important properties of a case are its hardness, ductility and depth. A microscopic scratch hardness method, such as employed in the Microcharacter, applied to a cross-section of the case gives an accurate measure of its hardness and depth. However, this method is so laborious and time-consuming that other methods have been sought. A diamond ball penetrator with variable depth of penetration and load, as exemplified in the Monotron, was found to be useful when proper choice of penetration depth was made. Likewise of interest is the comparison of spots on a single specimen, or a number of specimens in which the hardness-depth characteristics are similar, or on which an integrated hardness-depth effect is desired. This can be made with sufficient sensitivity by the use of the Rockwell $\frac{1}{16}$-inch ball and 100-kilogram load, care being taken to use a new standardized ball for each reading. As this is a practice not usual with the Rockwell machine, and as the readings are much higher than generally considered with the Rockwell B scale, the authors have chosen to call this the Rockwell Bx hardness.

Case ductility has been studied by the relation of load and type of failure produced with a 10-millimeter Brinell ball under variable loads. The macrographs Figs. 1 and 2 show typical brittle and ductile cases. These impressions were made with a 500-kilogram load. The cracking at the edge of the impression, as well as in the center, is very evident in Fig. 1 and indicates a comparatively brittle condition. The absence of cracks in Fig. 2 is a positive indication that the case will not spall readily.

The matter of case depth has been studied with the microscope. The problem was to find a reagent which would not only reveal the structure of the case but would also etch the material so that a line of demarcation would result corresponding to a definite and appreciable increase in hardness. Such a reagent has been developed by Mr. Vilella, of these laboratories. The correlation between case depth and etching effect is clearly shown in Fig. 3, in which a Microcharacter scratch is shown on an etched surface. The distinct and sudden change in width of the scratch at the edge of the etched band is illustrative of the action.

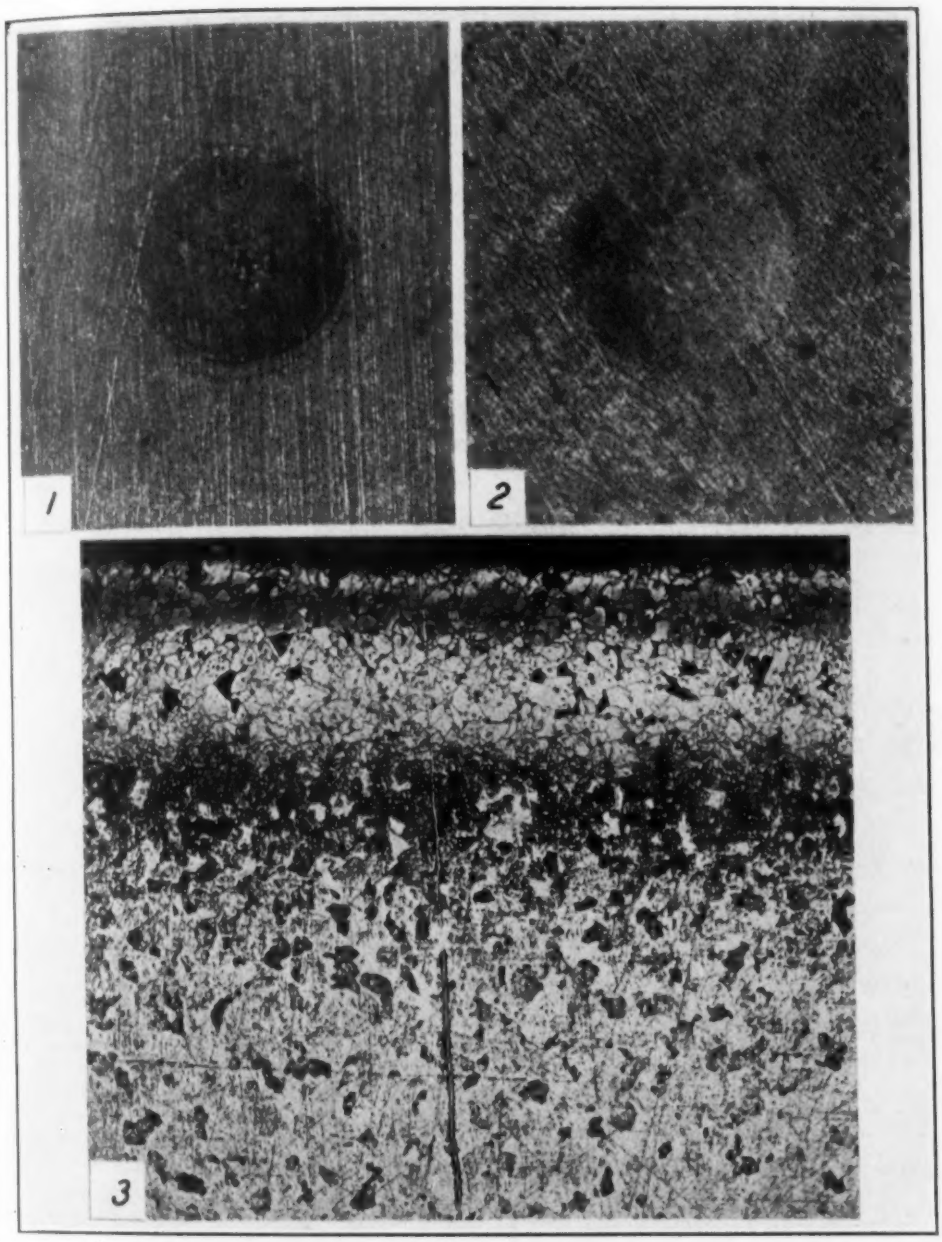


Fig. 1—Brinell Impression on Brittle Case. 500 Kilogram Load. × 15.
Fig. 2—Brinell Impression on Ductile Case. 500 Kilogram Load. × 15.
Fig. 3—Microcharacter Scratch on Nitrided Chromium-Aluminum Steel. × 100.

The etching procedure is as follows: Dissolve in 100 milliliters of water:

1.25 grams copper sulphate
2.50 grams cupric chloride
10 grams magnesium chloride
2 milliliters concentrated hydrochloric acid

Dilute to 1000 milliliters with 95 per cent ethyl alcohol. The ingredients should be carefully weighed and measured, as inaccuracies result in marked differences in the etching properties of the solution. Excessive copper sulphate diminishes color contrast, while increased cupric or magnesium chloride retard the copper deposition and impair the reagent's sensitivity. The specimen should be immersed in the reagent, polished surface down, as dropping the reagent on the surface results in very confusing edge effects. The quality of the etch can often be improved by repeated polishing and etching. Ten seconds' immersion is ample time to develop the case on either chromium-vanadium or chromium-aluminum steels.

Thus, the hardness, ductility and depth of the case are evaluated by means of the Microcharacter, Monotron, Rockwell Bx, Brinell 10-millimeter ball and Vilella etch.

Nitriding Without Packing Materials

The present study was carried out on chromium-aluminum and chromium-vanadium steels, the former being commercial material obtained from a well-known manufacturer of nitriding steels, the latter the minimum 0.4 per cent vanadium steel previously developed at the Union Carbide and Carbon Research Laboratories by the authors. A temperature of 460 degrees Cent. (860 degrees Fahr.) was used throughout on all nitriding, as general experience indicates that it results in a satisfactory case. On nitriding the above steels in ammonia at this temperature in the absence of packing materials a truly hard case is not produced in less than ten hours' nitriding time. Typical case structures are shown in the micrographs, Figs. 4, 5, 6 and 7, showing each of the steels as nitrided for 4 and 50 hours. These may be used as standards of comparison with the cases to be subsequently discussed.

Packing Materials as Accelerators

In considering possible packing materials with a view to accelerating the nitriding action, the first thought was to use materials which would react with or adsorb the hydrogen. This would allow a higher active nitrogen concentration at the steel surface. However, the packing material would soon become exhausted and would have to be frequently replenished. Accordingly, the authors pre-





Fig. 4—
Fahr. (460
Fig. 5—
Fahr. (460
Fig. 6—
Fahr. (460
Fig. 7—
Fahr. (460

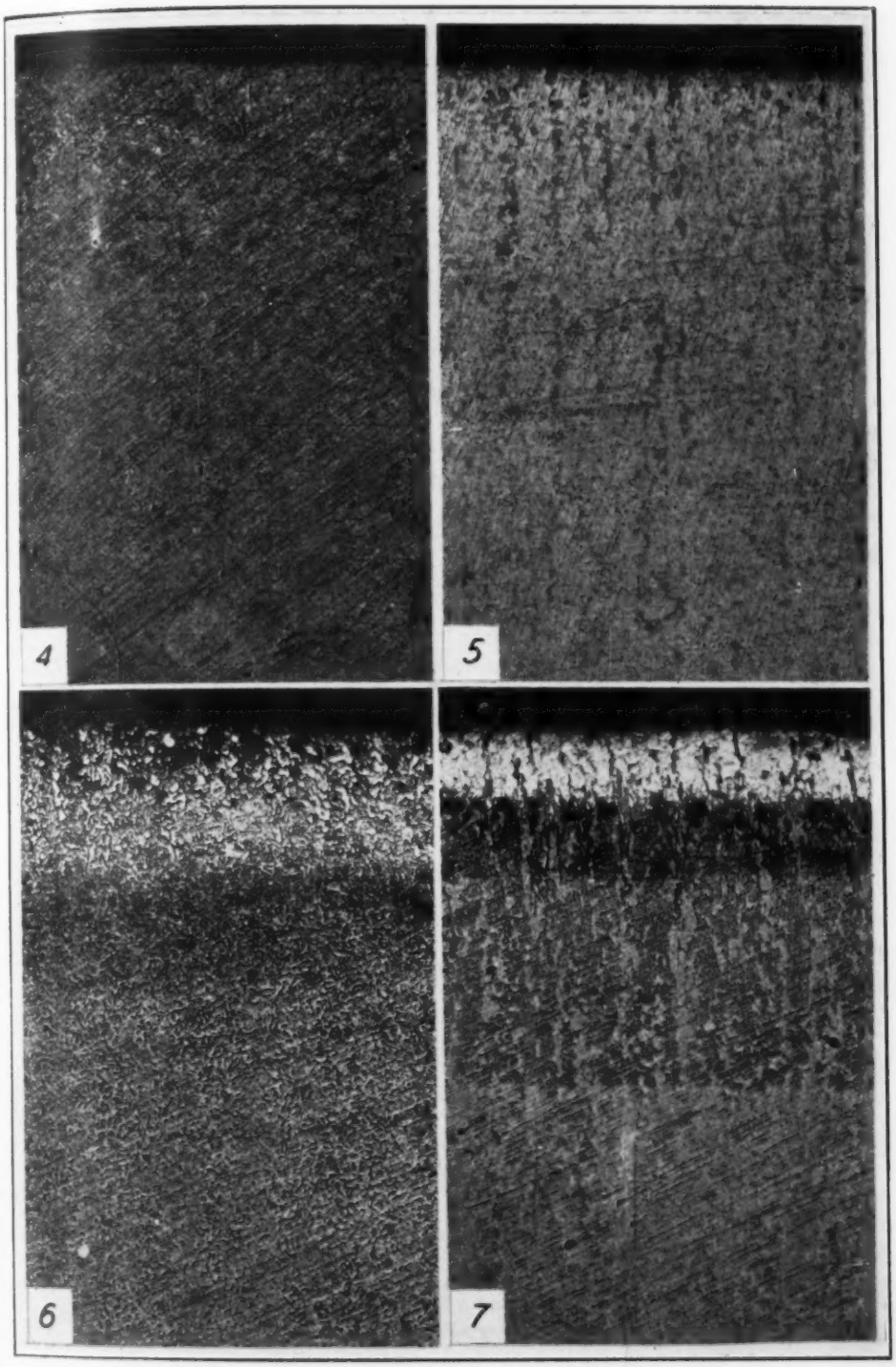
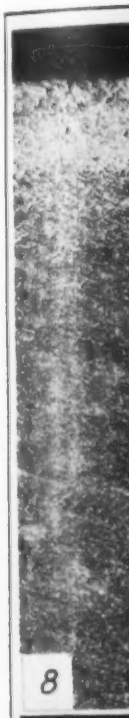


Fig. 4—Chromium-Aluminum Steel Nitrided 4 Hours in Ammonia at 860 Degrees Fahr. (460 Degrees Cent.) Etched with Vilella's Reagent. × 100.
Fig. 5—Chromium-Vanadium Steel Nitrided 4 Hours in Ammonia at 860 Degrees Fahr. (460 Degrees Cent.) Etched with Vilella's Reagent. × 100.
Fig. 6—Chromium-Aluminum Steel Nitrided 50 Hours in Ammonia at 860 Degrees Fahr. (460 Degrees Cent.) Etched with Vilella's Reagent. × 100.
Fig. 7—Chromium-Vanadium Steel Nitrided 50 Hours in Ammonia at 860 Degrees Fahr. (460 Degrees Cent.) Etched with Vilella's Reagent. × 100.

ferred to study materials which are apparently unchanged during the nitriding cycle, and which may, therefore, be used for indefinitely long periods. The packing materials, or accelerators, herein described may be broadly divided into two classes, nonmetallic and metallic. In either type the state of division is important. In general, the finer the state of division the more satisfactory is the acceleration.

The action of the nonmetallics is best illustrated by magnesia. This is used in the form of powder, through 275 mesh. The steel is completely surrounded by the magnesia, and ammonia is passed through the container in such a manner that it comes in contact with the work. It is only necessary that the steel be covered by a light layer of magnesia. Heating the nitriding steels in this way for four hours at 460 degrees Cent. (860 degrees Fahr.) results in a hard case. Treatment for 50 hours under the same conditions results in a case of much greater depth than would be produced in the same time in the absence of packing materials. These cases are shown in Fig. 8, 9, 10 and 11, and are directly comparable with Fig. 4, 5, 6 and 7. The decided improvement obtained by the use of the packing material is obvious. Moreover, corrosion tests on the four-hour cases show them to be satisfactory from this point of view. Thus, if a light but hard corrosion resistant case is desired, a nitriding cycle is possible which is shorter than hitherto available; this, in addition to the fact that the use of packing materials reduces the nitriding time for a given depth of case. This reduction in time may be somewhat offset by the increased time necessary to heat the container if a large amount of packing material is employed. Even in this case, however, the packing material is warranted, due to other advantages to be discussed later. Although magnesia seems to be the best of the inert materials, others such as silica flour, lamp black, etc., have been used with success.

Because of the insulating effect of the packing materials above mentioned the use of metal packings was investigated. Results similar to those produced by the magnesia were obtained. Copper seems to be the most satisfactory of the metallic materials. Table I shows hardness as a function of time, with and without copper packing, on both chromium-aluminum and chromium-vanadium steels in the heat treated and non-heat treated conditions. Two penetrating depths were used in the measurements, so that these figures are also an indication of the depth of case. A glance at these data shows the ef-



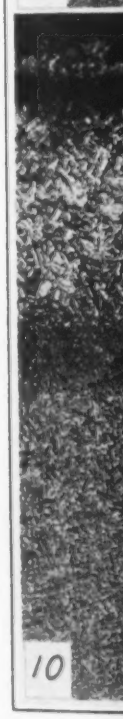


Fig. 8—
Degrees Cen
Fig. 9—
Degrees Cen
Fig. 10-
Degrees Cen
Fig. 11-
Degrees Cen
Fig. 12—
Degrees Cen
Vilella's Rea

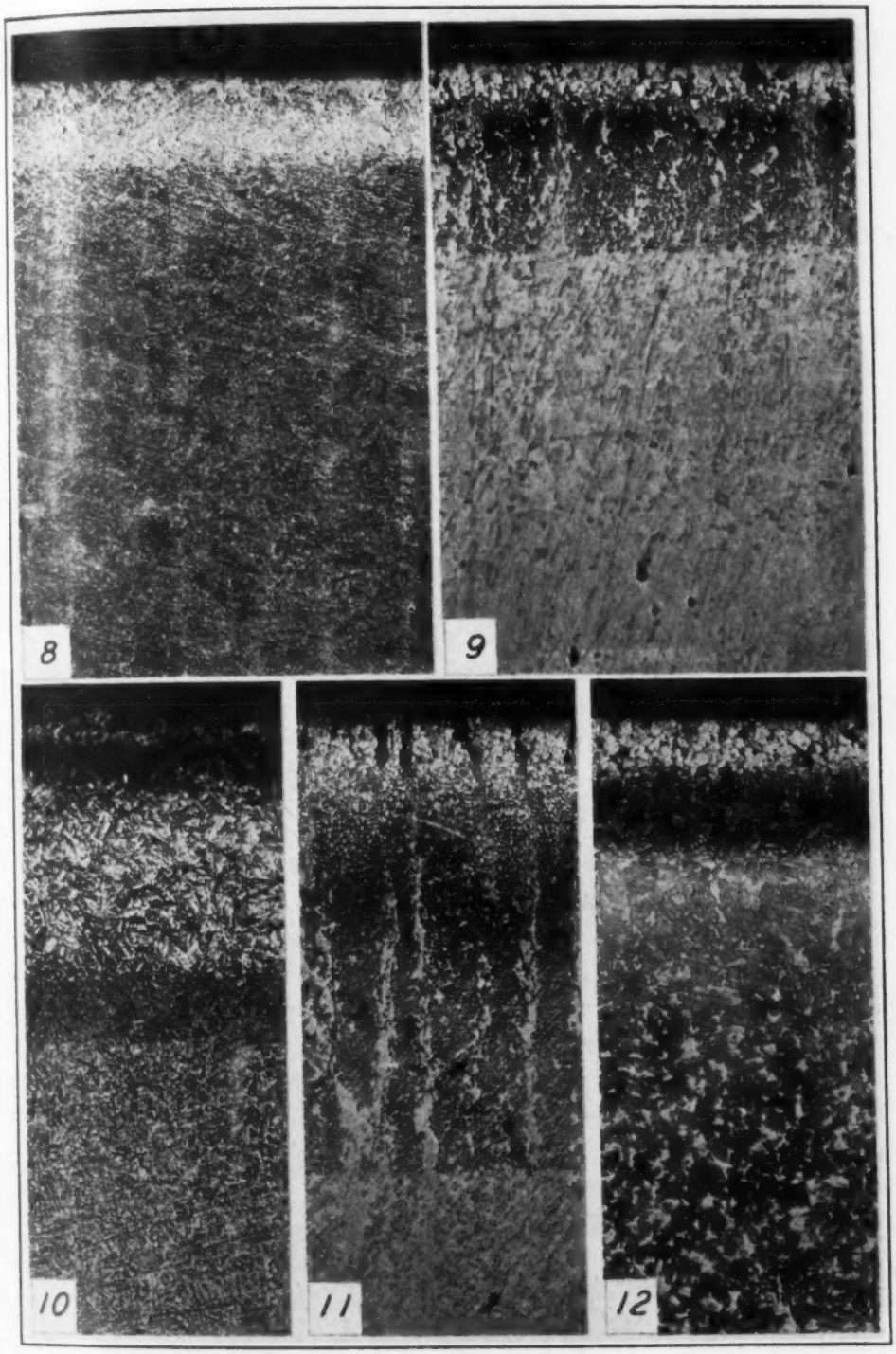


Fig. 8—Chromium-Aluminum Steel Nitrided 4 Hours at 860 Degrees Fahr. (460 Degrees Cent.) in Ammonia with Magnesia Accelerator.
Fig. 9—Chromium-Vanadium Steel Nitrided 4 Hours at 860 Degrees Fahr. (460 Degrees Cent.) in Ammonia with Magnesia Accelerator.
Fig. 10—Chromium-Aluminum Steel Nitrided 50 Hours at 860 Degrees Fahr. (460 Degrees Cent.) in Ammonia with Magnesia Accelerator.
Fig. 11—Chromium-Vanadium Steel Nitrided 50 Hours at 860 Degrees Fahr. (460 Degrees Cent.) in Ammonia with Magnesia Accelerator.
Fig. 12—Chromium-Vanadium Steel Nitrided 8 Hours at 860 Degrees Fahr. (460 Degrees Cent.) in Ammonia with Copper Gauze Accelerator. All Specimens Etched with Vilella's Reagent.

Table I

Monotron Pressure Readings

Steel	Time	NH_3 only As rec'd	NH_3 only Ht. Treat.	NH_3 Plus Accelerator As rec'd	NH_3 Plus Accelerator Ht. Treat.
		1/5000 inch Penetration			
Cr-Al	4	6	6	10	10
	20	11	9	12	13
Cr V	4	7	8	8	10
	20	8	8	10	12
		1/1000 inch Penetration			
Cr-Al	4	17	23	25	30
	20	30	34	45	50
Cr-V	4	22	28	28	35
	20	28	35	41	49

ficacy of copper as an accelerator. The state of division, however, is again important. Very fine powder has never been tried since it was believed that its insulating effect would more than balance any increased usefulness as an accelerator. Fine chips, gauze, or filings have been used with success. Copper, brass and aluminum were the most satisfactory metals used, but in the case of aluminum the accelerating action was only effective on the aluminum-bearing nitriding steel. A typical case obtained with the use of 15 mesh copper gauze in eight hours' nitriding time is shown in Fig. 12.

Extremely interesting results were obtained by the use of prenitrided packing materials. Chips of chromium-aluminum and chromium-vanadium steels were nitrided for long periods so that the case extended through the chips. This nitrided material was then crushed to a powder and used as packing material. When the powder had been prenitrided at temperatures below 580 degrees Cent. (1075 degrees Fahr.) and chromium-vanadium steel was packed in chromium-vanadium prenitrided powder, or chromium-aluminum steel was packed in prenitrided chromium-aluminum steel powder, very satisfactory results were obtained. These were almost identical with the results obtained by the use of magnesia. However, aluminum steels packed in prenitrided chromium-vanadium turnings failed to show any improvement in case whatever, and vice versa. Both types of steel may be satisfactorily nitrided simultaneously in a mixture composed of equal parts of the two prenitrided materials. When the turnings were nitrided at temperatures above 600 degrees Cent. (1110 degrees Fahr.) no accelerating action was noted. This phenomenon is most interesting, and with further study should lead to increased comprehension of the nitriding mechanism.

Table II

Rockwell Bx Obtained in Various Locations in Nitriding Box

	Without Packing Materials 1 Exit Tube No. 1	Without Packing Materials 8 Exit Tubes No. 2	With Packing Materials 1 Exit Tube No. 3
A	123	126	128
B	124	127	127
C	121	127	127
D	125	127	128
E	123	126	127
F	124	126	128
G	124	126	127
H	126	127	127
I	127	123	126
J	126	124	127

Letters A to D represent the four top corners, E to H the bottom corners, and I and J the top and bottom center locations in the nitriding box. Ammonia entered above point I.

Uniformity in Nitriding

The second problem mentioned in the opening paragraph is the matter of case uniformity from heat to heat and on specimens packed in a single container in any given heat. Experiments have shown clearly the remarkable effect of packing material on uniformity. In Table II column 1, Rockwell Bx hardness figures are given for heats made in a container 25 by 20 by 15 inches, with the ammonia introduced at one point near the center of the top, and the exhaust gases removed at a single point at the bottom center of one end. The lack of uniformity in hardness in some of the specimens is very evident. Other runs were made, in which the ammonia was introduced at the same point, but removed at eight points corresponding to the corners of the box, special copper tubes having been arranged so that uniform effusion of the gases occurred at the corners. In spite of this precaution lack of uniformity resulted, as shown in the second column of Table II and all efforts to obtain uniform nitriding in the container in this manner failed. However, by covering the specimens with magnesia and having the gas flowing out of the various corners, a case was produced equal to the best obtained throughout the box. By packing the specimens in magnesia and using a single inlet and outlet for the gas, remarkable uniformity was also obtained, as shown in the third column of Table II.

The results actually shown here are only one set from a great many heats, and this phenomenon has been repeatedly checked. Moreover, on nitriding heat after heat on a 20-hour cycle with the use of magnesia or equivalent packing material extremely uniform

results have been regularly obtained. It is particularly interesting that these results are independent of the type of container used or of the condition of its inside surface. It is still further significant that experiments indicate that the type of case obtained when using a packing material is independent of the dissociation of the gas within very wide limits, so that a careful control of this factor is unnecessary when packing materials are used.

Conclusions

It is expected that the use of packing materials as here indicated will find ready commercial applications due to the following advantages:

1. Finely divided magnesia, copper gauze, chips or filings, as well as other materials herein described produce increased depth of case in a given time of nitriding in ammonia, when used as packing materials.
2. The case produced in four hours in ammonia by the use of these packing materials is corrosion resistant to salt spray.
3. The uniformity of the process is insured by the use of these packing materials.
4. Packing materials may be used with equal success on chromium-aluminum or chromium-vanadium steels.

Acknowledgment

The authors wish to express their appreciation of the interest and stimulation given by F. M. Becket, president, and J. H. Critchett, vice-president of the Union Carbide and Carbon Research Laboratories, as well as the general assistance rendered by their colleagues at the Laboratories, and to mention particularly the work done by J. Lamont, C. Kluge and C. Hergenhan. Special credit is also due J. Vilella for the development of the etching process and preparation of the photomicrographs.

DISCUSSION

Written Discussion: By Robert Sergeson, metallurgical department, Central Alloy Division, Republic Steel Corporation, Massillon, Ohio.

Messrs. Kinzel and Egan have shown new and very interesting data and the methods mentioned certainly warrant further study as to practical application. Not having conducted similar work, the writer wishes to

ask the authors some questions that have come to mind in reading this paper.

First—Would not the time required in packing and bringing container to temperature be excessive for practical nitriding in large containers? Also would not the length of time in cooling likewise be excessive? Writer believes that owing to the low temperatures used in nitriding that packing materials will not be successful for large containers but may be used in small containers.

Second—Why have the authors selected 860 degrees Fahr. for their nitriding temperature when the commercial nitriding temperatures are much higher, 950-1000 degrees Fahr? Have they experimented these higher temperatures and, if so, with what results?

Third—In comparing the case depth and hardness of the four hour runs, writer believes that the samples placed in the packing material are actually being exposed to nitriding conditions for a longer period than four hours. This is due to fact that container must be brought up to the nitriding temperature very slowly owing to the sluggishness of heat passing through the packing material. The fact that the 50-hour runs do not show such a marked difference in case depth between the packed and not packed runs appears to substantiate this belief.

Written Discussion: By John P. Walsted, instructor in physical metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

The paper by Mr. Kinzel and Mr. Egan comes at a very opportune moment. Uniformity of nitriding and dependability of the nitriding process are of greatest importance. Any decrease in the nitriding time is an improvement that will be welcomed by all who are trying to do nitriding on a commercial scale.

The use of copper as a packing material must be undertaken with caution. Copper in the nitriding furnace will cause the formation of a blue crystalline deposit at the outlet end of the ammonia system. This deposit is liable to cause stoppage of the gas flow. This difficulty has been encountered in our work at M. I. T. when using copper trays.

I would like to ask if the authors have tried filings of iron or low carbon steel. Cast iron chips are easily obtained in a fine state of subdivision and in addition contain much carbon. The high percentage of carbon should aid in eliminating the hydrogen.

The matter of case ductility is of greater importance than many realize. All methods of test have serious disadvantages. It is almost impossible to devise a test that will not be influenced to a great extent by case depth and core hardness. The Brinell test used by the authors fixes an arbitrary limit between brittle and tough cases. Dr. Homerberg and I are using a Brinell machine for this test without an arbitrary limit. We place the specimen under the ball and gradually increase the load until the case cracks. The load is then read giving us comparative values of ductility. The cracking of the case can be readily detected if the test is conducted in a moderately quiet room. In this way we get readings as low as a 150-kilogram load and as high as 3000-kilogram load. The test is undoubtedly

crude but the results give us a great deal of information not otherwise obtainable. Moreover, duplicate readings check surprisingly well.

W. J. MERTEN:[1] Mr. Kinzel's interesting investigation on nitriding in packing material brings to my mind a similar experiment which I conducted for some other reason, however. I wrapped a piece of nitriding steel into aluminum foil and obtained surprisingly good results and in a short time of exposure the improvement was quite noticeable in greater degree of hardness, as well as improved surface conditions over the ordinary nitriding process. I did not go into this more thoroughly. Mr. Kinzel's investigation certainly does prove that there are common materials, which, when applied judiciously in the nitriding practice, do improve the case with reference to hardness as well as depth. Along with it the surface finishes remain perfect which obviously is of value in processing finish machined parts. Mr. Kinzel certainly is to be congratulated upon the very excellent results that he has obtained for this investigation and upon his valuable contribution to the art of nitriding.

J. H. HIGGINS:[2] Mr. Kinzel made mention of unsatisfactory methods of testing the ductility of the case. I would like to advise of the method we use at our plant. Perhaps he may find it to some advantage. We prepare ½-inch specimens, 8 inches long, polish and nitride, then space them carefully on the machine, applying pressure as in regular bend testing, and recording the strength required to fracture the case. Very definite ductility tests can be made in that manner.

P. A. E. ARMSTRONG:[3] I did not intend to take part in this discussion. Down in New York, Mr. Kinzel replied on behalf of a question that I asked someone else. He said, you cannot nitride in the presence of carbon. If there is any carbon on the surface of the metal it will not take up nitrogen. I am very pleased to say that he has confirmed what I told him. "Come and see me and you'll know how it's done." I noticed in his paper it can be done. Incidentally mixing up carbon dust with nickel granules produces about the same results that Mr. Kinzel has so admirably shown. The parts may be packed in that mixture or they may be put in between wads of the mixture inside of gauze. I have tried both copper and nickel gauze and find out that it works out very well indeed.

D. LEWIS:[4] I would like to ask Mr. Kinzel what is the composition of his chromium-vanadium steel. He does not actually state its composition in the paper. I am very much interested in the use of copper. He speaks of using copper tubes for inlet and exit tubes for the ammonia. I would like to ask whether he has had experience of copper in the nitriding box, whether it is unaffected by the ammonia, whether a box might be made out of the copper or whether a box might perhaps be copper lined. Also with

[1]Metallurgical engineer, Westinghouse Electric and Manufacturing Company, East Pittsburgh.

[2]Material engineer, Camden Forge Co., Camden, N. J.

[3]Consulting metallurgist, 875 Park Ave., New York City.

[4]Research engineer, American Chain Co., Bridgeport, Conn.

reference to the time required to get heat through a magnesia filled box. Has he tried the possibility of coating the parts to be nitrided with a paste of magnesia?

Authors' Closure

In regard to Mr. Sergeson's discussion, the first point he brought up was the time in packing and cooling. That is a question that will have to be settled later. We cannot tell how serious that is from laboratory experience. However, we can say that it is not necessary to fill a box completely with material for a few odd pieces of nitrided steel. As long as the parts to be nitrided are covered with a layer of inert material acceleration and uniformity are achieved. In our work conditions are such that insulating difficulties do not trouble us.

Regarding the use of 460 degrees Cent. as the nitriding temperature, we chose that temperature for several reasons. Our previous work on the chromium-vanadium steel had been done at that temperature, and we believe that it is the optimum temperature for chromium-vanadium steels. We have gone to a higher temperature and the same comparative results are found. We have not done that in as many cases as we have nitrided at the lower temperature so we did not see fit to include those results in the report.

It is Mr. Sergeson's belief that possibly the 4-hour treatment was a good deal longer than four hours. We have an arrangement whereby the container is flushed with ammonia and then put into the hot furnace. Thermocouples show that in the case of the small containers used here the work comes up in a quarter of an hour. There is a possibility, then, that it was four hours plus something less than a maximum of a quarter of an hour. Regarding the difference between the effect we get with the short and long time runs, that difference is actually real. I think it is tied up with the two phases involved in the nitriding. First the nitrogen is put in at the surface and then it is diffused inwards. One or the other is a limiting reaction. At the start of a nitriding run for the first hour or two the rate of nitrogen entering at the surface is the limiting factor and it will diffuse as fast as but not faster than the rate at which it enters if the inert material is used. If 50 or 90 hours are used the diffusion as greater depths are reached will decrease logarithmically and a good deal of advantage is lost. The inert material is intended primarily for a cycle of about 20 hours because in that time, using inert material, those case depths result which are generally needed for commercial applications as we know them today.

Regarding Mr. Merten's discussion, we are very much pleased indeed to see that there is corroborative evidence for our work. We describe gauzes that correspond to the foil that Mr. Merten used and this serves to bring out more than ever the need for further work on the whole nitriding process, and to emphasize the excellent possibilities of making marked improvements. We wish to thank Mr. Merten for his discussion.

Mr. Higgins mentioned a method of measuring ductility. By a bend test or a tensile test one can observe when a crack first appears. We are strong advocates of the bend test and it seems to us that Mr. Higgins is

using the bend test and measuring the ductility in the same way as we have done in the tensile test. The only objection to that method is the same one that applies in the tensile test, namely the difficulty in determining when an opening in the case first occurs. In the tensile bar, for example, those rings actually appear in the microscope long before one can see them with the naked eye. The method in general, however, is a very good qualitative measure. It undoubtedly meets present needs. However, it still has certain limitations as a quantitative test.

Regarding Mr. Armstrong's discussion, I believe that I have been misquoted on this subject. In the meeting that we had in New York City someone wanted to know if one could nitride by adding acetylene to the ammonia and I said we could not, because of clogging of the tubes. I am sure that Mr. Armstrong and I are in entire agreement on the efficacy of carbon as a packing material, although we may differ as to the mechanism involved.

Regarding the discussion by Mr. Lewis, a 1.5 per cent chromium, 0.5 per cent vanadium content helps in both core and case properties, and is generally beneficial. The carbon can be made to suit. In this particular steel it was 0.27 per cent. The carbon range can be set and then the minimum vanadium content determined, because satisfactory nitriding depends on effective vanadium. The higher the carbon the more vanadium necessary.

Regarding the copper tubes, we used them because we planned only a few runs and could handle them carefully. We found that in nitriding copper gets very brittle. It may be possible to line a container with it and handle it in such a way that brittleness is immaterial.

Regarding the total time effect, one can cover the pieces with a light layer and we have used not only paste but various other kinds of mixtures which could be applied to the surface. These are satisfactory but there is the nuisance of application which depends on the particular problem.

Mr. Walsted's discussion was distinctly pertinent. We have not had any difficulty due to stoppage of gas flow when using copper as a packing material, although on occasion we have noted a very slight coloring on the glass of the exit tube. It may be that the difference between our practice and Mr. Walsted's, namely the drying of the ammonia in all our tests, eliminates the difficulty in question. In all of our runs, ammonia is bubbled through glycerine or ethylene glycol before being passed into the nitriding container.

Iron filings and low carbon steel have been tried as packing materials without success. Cast iron chips have also been tried, but the improvement in nitriding obtained thereby has not been sufficient to warrant the recommendation for this usage in view of the marked effect of the copper or the magnesia.

We agree heartily with Dr. Walsted regarding the importance of ductility, and read with great interest of the sonic method of determining the end point of the cracking test. We have attempted to use this method since hearing from Dr. Walsted, and have even gone to the extent of applying the stethoscope to the specimens in question. However, we found in all instances that the case cracks before any indication of this cracking is af-

forded by sound, and it is our belief that the sound occurs either with brittle cases only or at the time the case breaks through over the entire area under test. In either case, we do not believe that the end point thus afforded by the sound is any more reliable than that which we get by examination with the stereobinocular. We are, however, planning to carry on further work along these lines, as we appreciate the inadequacy of the present tests.

FURTHER INVESTIGATIONS IN NITRIDING

By R. Sergeson and H. J. Deal

Abstract

The authors have grouped the work reported in this paper into three sections. Section I is given over to a discussion on nitriding containers. The Section II covers corrosion tests on the nitrided surface. The effect of acids, bases, salt spray, salt water, tap water, oils, atmosphere, etc., are recorded. In Section III the subject of denitriding is discussed, giving the methods and results.

Section I

THE subject of nitriding containers is indeed an important one. In the commercial nitriding field there are many different types now in use. The question as to which is the best is still a matter of opinion. This is undoubtedly due to the comparatively short time that commercial nitriding has been practiced. The authors wish to give their experience and suggestions, hoping others will add further in way of discussion. In attacking this problem, only the materials of construction and methods of sealing will be discussed.

Materials of Construction

Plain Carbon Plate Steel: The earliest and in fact some of the present day containers were made from carbon plate steel. Experience with welded containers of this material has not been satisfactory for the following reasons. In the nitriding process, the carbon steel nitrides and in time disintegrates. After 500 hours of nitriding in such containers it is noticed that an increased rate of ammonia flow is required to maintain the required dissociation (which should be 20 to 30 per cent). The longer the container is used, the greater the rate of ammonia consumption, the lower the surface hardness and the shallower the case until a point is reached where regardless of rate of ammonia flow, unsatisfactory nitriding results. An explana-

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. Of the authors, who are associated with the metallurgical laboratories of the Republic Steel Corp., Massillon, Ohio, Robert Sergeson is a member of the society. Manuscript received June 7, 1930.

tion of this is offered. As the inside walls of the container become nitrided they act as catalytic agents, breaking up or dissociating the ammonia on the walls. This has the effect of increasing the dissociation and can be counteracted by increasing the rate of ammonia flow. However, as nitriding takes place only when the nitrogen is in the nascent state, it is necessary that the ammonia break up on the work being nitrided. If the ammonia breaks up on the container walls it is probable that the nitrogen will become molecular and inert before it reaches the work. Thus the increase in flow really produces a diluting effect and a considerable part of the nitrogen cannot be used owing to its becoming molecular and inert on the walls, hence less nitrogen is available for nitriding. If the ammonia flow is not increased, the dissociation will become high, which produces a soft case due to the reversing of the reaction by the liberated hydrogen. It would appear therefore that an ideal container would be one that is inert to the action of ammonia at the nitriding temperatures. This has been aimed at and will be discussed under enameled containers.

Stainless Irons

Both the high chromium (0.10 C—16.0 Cr) and the high chromium-nickel (0.10C—18.0 CR—8.0 Ni) have been used for nitriding containers. Up to 1000 hours these containers have given good results. Above this, however, the same trouble arises as mentioned under the carbon steel containers, namely the ammonia consumption must be increased and a falling off in surface hardness and case depth are observed. Another factor has been checked. Nitralloy steel drillings nitrided in a container when new show an approximate nitrogen content of 8.0 per cent. After 2000 hours Nitralloy drillings nitrided under same temperature and time conditions show only a 4.0 per cent nitrogen content. This explains the lower hardness and lower corrosion resistance. Sand blasting the nitrided surface of the container gives temporary relief. Another cure has been suggested by R. G. Roshong[1] which is to heat the container to 1500 degrees Fahr. while passing ammonia through. This again gives only temporary relief.

Monel Metal

This metal with low manganese content has been used by the

[1]Nitriding Symposium—A. S. S. T. Sept. 13, 1929, p. 220.

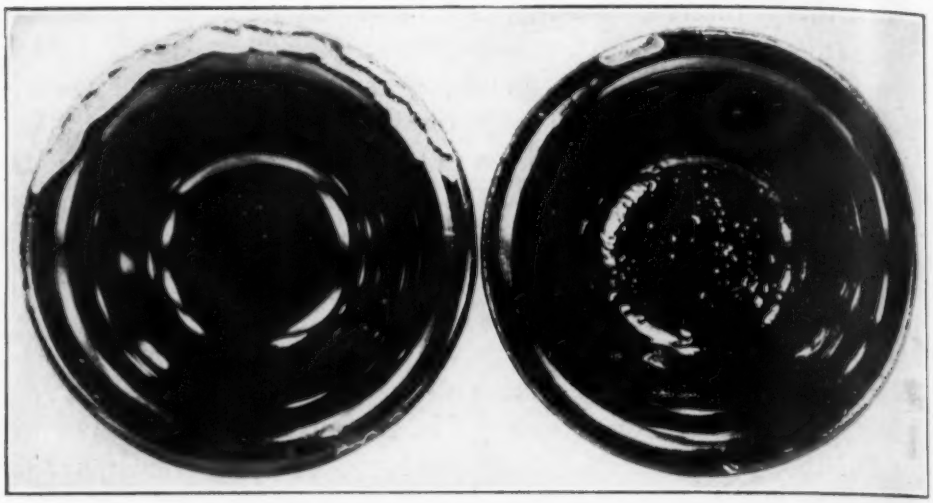


Fig. 1—(Left) Chemically Resistant Enamelled Pot Heated in Nitriding Atmosphere at 1050 Degrees Fahr.
Fig. 2—(Right) Enamelled Pot (Hard Enamel) Heated in Nitriding Atmosphere at 1050 Degrees Fahr.

authors up to 1000 hours and has shown no tendency to nitride. The dissociation has remained constant with given rate of flow and temperature. Check runs on nitrided Nitralloy steel drillings show that the nitrogen content is the same on early and recent runs. Satisfactory nitriding is still being obtained. This metal shows much promise but a longer period of service must be obtained to determine its worth.

Enamelled Surfaces

Having a stainless iron container (0.10 C, 18.0 Cr, 8.0 Ni) all fabricated at the time this analysis was found not to be entirely satisfactory, it was decided to enamel the inside surface. Preliminary work had already been completed on a laboratory scale to justify this step. Enamelled tests of both carbon steel, enamelling iron and stainless iron were subjected to the various nitriding conditions and temperatures. All hard enamels were excellent at 950 to 1000 degrees Fahr. in that they gave no signs of bubbling, flaking or creeping. From 1050 to 1200 degrees Fahr. most of the enamels flaked or bubbled. Fig. 1 shows a chemical resistant enamel which stood up to 1050 degrees Fahr. Fig. 2 shows an ordinary hard enamel which was satisfactory at 1000 degrees Fahr. but which bubbled badly at 1050 degrees Fahr. Further work is being done on enamels to withstand temperatures up to 1200 degrees Fahr. As the usual

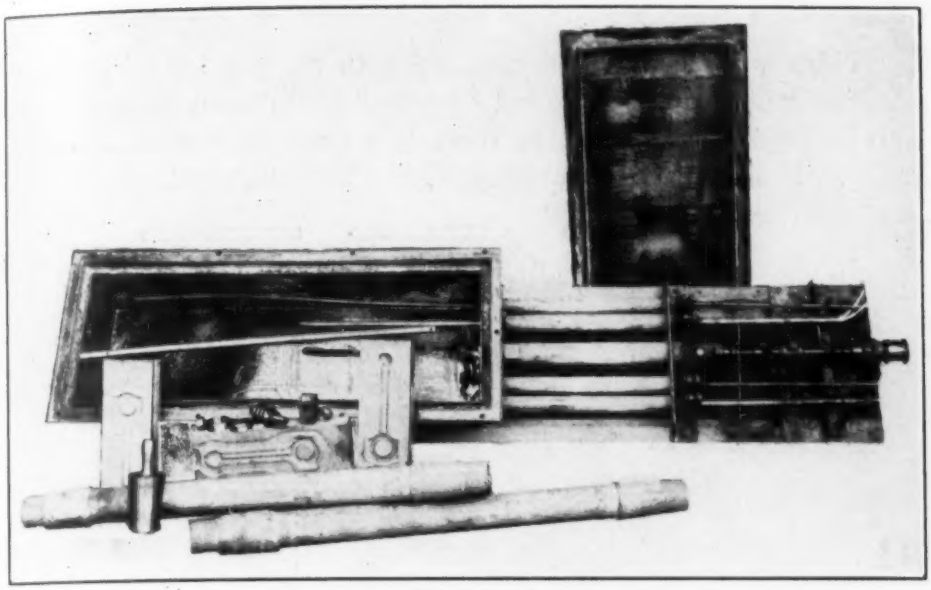


Fig. 3—Enamelled Nitriding Container. 18 Inches Wide, 12 Inches Deep and 52 Inches Long (Inside Dimensions). Uses Eutectic Tin and Lead Seal.

nitriding temperatures are not above 1000 degrees Fahr., the stainless iron container was sand blasted and enameled with the ordinary hard enamel. It was hoped that the enamelled surface would be inert to the influence of the ammonia gas under the nitriding conditions. Fig. 3 shows a photograph of this container after 700 hours of service.

The bottom of the container and under side of the lid show creeping has taken place due to expansion of the metal. The side walls are perfect. Thus far excellent results have been obtained. Again a longer test is required. It is quite probable that a glass-coated container would give good results and merits a trial. Carbon steel containers enamelled are now being tested.

Other Containers

In Germany the nitriding containers are made from N. C. T. 3 analysis (approximately 0.15 C, 25.0 Cr, 20.0 Ni). This is an expensive alloy and its merits not proved. Nichrome should prove satisfactory as heating elements of this material exposed to nitriding conditions for over 2000 hours appear unaffected. Again, however, this alloy is expensive.

Methods of Sealing Containers

Those who have been confronted with the problem of sealing a nitriding container have selected a number of different types of seals. It is the purpose here to list these types and briefly discuss their merits. In Fig. 4 the various types have been sketched and lettered.

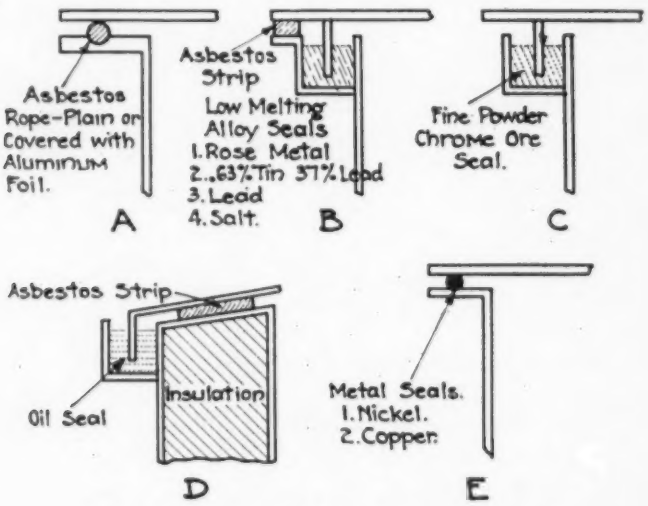


Fig. 4—Sketches of Various Types of Seals.

Type A represents the first type used, in which plain asbestos or asbestos covered with aluminum foil were employed for the sealing medium. This type required many bolts to insure a gas tight container. Such was difficult to obtain and work removed from these containers was invariably colored, indicating seal had not remained gas tight and air had entered. Type E is similar in that copper, aluminum or nickel in wire form is used in place of the asbestos. On larger containers the number of bolts required to insure a tight seal means considerable time and labor must be spent. Hence this type of seal has not been highly satisfactory.

The B type of seal shows the present trend to insure a perfectly gas tight container. In this method, the seal is a low melting alloy and is liquid during operations. Rose Metal (50.0 Bi, 25.0 Sn, 25.0 Pb) is satisfactory on small installations if protected by graphite and the asbestos auxiliary seal, from oxidation by furnace atmosphere. The less expensive low melting alloys, eutectic tin 63 per cent and lead 37 per cent or lead give better seals for larger containers. Salt has not been investigated thoroughly but offers possibilities. It should be mentioned that the low melting metal alloys

attack Monel metal, hence cannot be used with this metal. The attack causes brittleness and disintegration caused by intercrystalline penetration.

In Type C, a depth of 3 inches of finely divided chromium ore (150-mesh) has been found to give satisfactory results on a laboratory scale and merits further investigation.

Where the seal may be made outside of the heating zone, Type

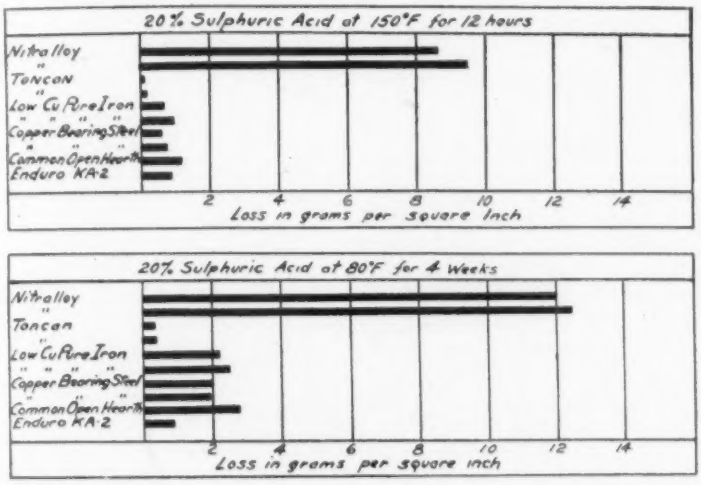


Fig. 5—Chart Showing Resistance to 20 Per Cent Sulphuric Acid Hot (150 Degrees Fahr.) and Cold.

D seal has worked satisfactory with an oil seal. Care, however, must be taken that a high enough flash point oil is used to prevent any oil from distilling into the nitriding chamber. Should oil distill into the nitriding chamber, the work will be colored and is apt to be soft.

Conclusions

For longer life of nitriding containers and low initial cost, the carbon steel container enamelled on inner surface presents excellent possibilities. Monel metal or Monel metal-lined containers, although more expensive, present a second promising material for nitriding containers.

Of the methods of sealing the containers, the liquid seals present the most efficient; low melting metal alloys should be used where seal is subjected to nitriding temperature and oil where the seal is outside and may be kept cool. A deep seal of powdered chromium ore has shown much promise.



Fig. 6—Photograph of Nitralloy Samples After Immersion in 20 Per Cent Sulphuric Acid at 150 Degrees Fahr. for 12 Hours.
Fig. 7—Nitralloy Specimens After Immersion in 20 Per Cent Sulphuric Acid Cold for 4 Weeks.

Section II

Although the nitrided surface is most noted for its hardness and resistance to wear, another property of perhaps equal importance is its corrosion resistance under many conditions. A study of various corrosive agents and their effect on the nitrided case have been made in our laboratory and observed in service. These tests will be described in a somewhat tabulated manner.

To determine the relative corrosion resistance of the nitrided case, Nitralloy G (0.34 per cent carbon) 0.715 inch in diameter by 4 inches long test samples were used. These samples were nitrided for 90 hours at 975 degrees Fahr. developing a hardness of 980 Vickers Brinell with a case depth of 0.030 inch. The classification of these tests is based on gram loss per square inch of exposed surface area.

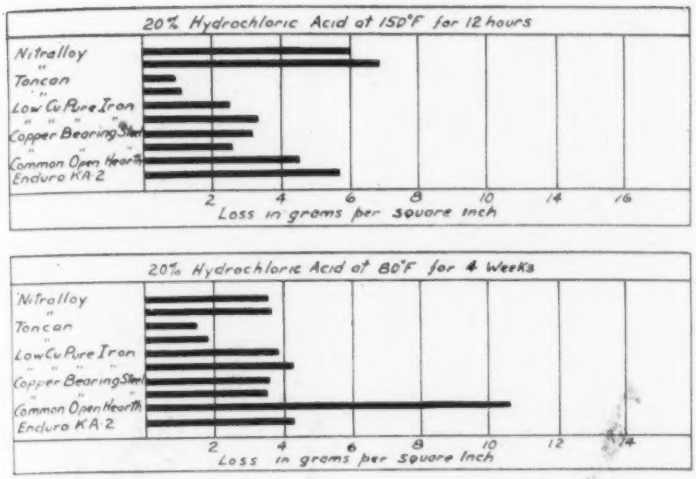


Fig. 8—Chart Showing Resistance to 20 Per Cent Hydrochloric Acid Hot (150 Degrees Fahr.) and Cold.

A—20 Per Cent Sulphuric Acid at 150 Degrees Fahr. and Cold

Weighings were made and solutions renewed every two hours for six periods in the hot test. The cold test was run for four weeks, the solution being renewed and weighings made each week. The resistance of the Nitralloy case has been plotted in chart form and compared to some of the corrosion resistant materials found on the commercial market. Fig. 5 shows charts of hot and cold tests while Fig. 6 and Fig. 7 show photographs of the Nitralloy samples after above tests.

In both hot and cold tests the points of weakness seem to be the ends of the samples. At this point the attack is similar to that of unnitrided samples, having penetrated deeply. Sulphuric acid (20 per cent), hot or cold, shows severe attack on the nitrided specimens.

B—20 Per Cent Hydrochloric Acid at 150 Degrees Fahr. and Cold

Two samples were used for each test. The hot test was run for twelve hours, weighings made and solutions renewed every two hours. The cold test was run for four weeks, the weighings made and solutions renewed each week. Results of tests are shown in Fig. 8 in which again the Nitralloy case is compared with other

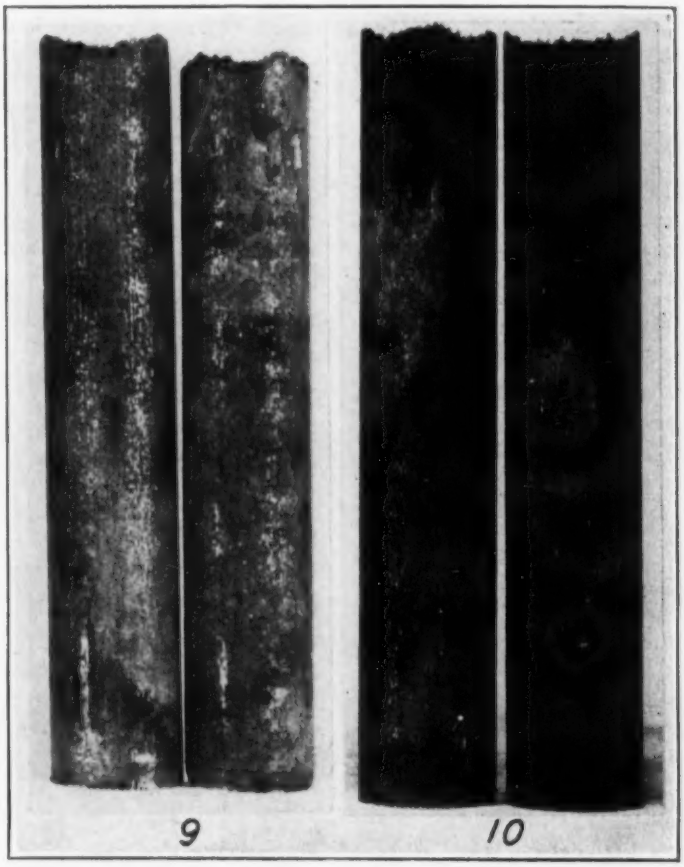


Fig. 9—Photographs of Nitralloy Samples After Immersion in 20 Per Cent Hydrochloric Acid 105 Degrees Fahr. for 12 Hours.

Fig. 10—Nitralloy Specimens After Immersion in 20 Per Cent Hydrochloric Acid Cold for 4 Weeks.

well known products. In Fig. 9 and 10, photographs of the Nitralloy tests are shown.

Nitralloy is severely attacked in both the hot and cold 20 per cent hydrochloric solutions. The attack is centered on the ends of the test samples as in the 20 per cent sulphuric acid tests.

C—Crude Oil at 150 Degrees Fahr. and Cold

The hot solution was held at 150 degrees Fahr. for 100 hours and weighings were made and solutions renewed every 25 hours. The cold test was run for four weeks. Weighings were made and the solution renewed each week.

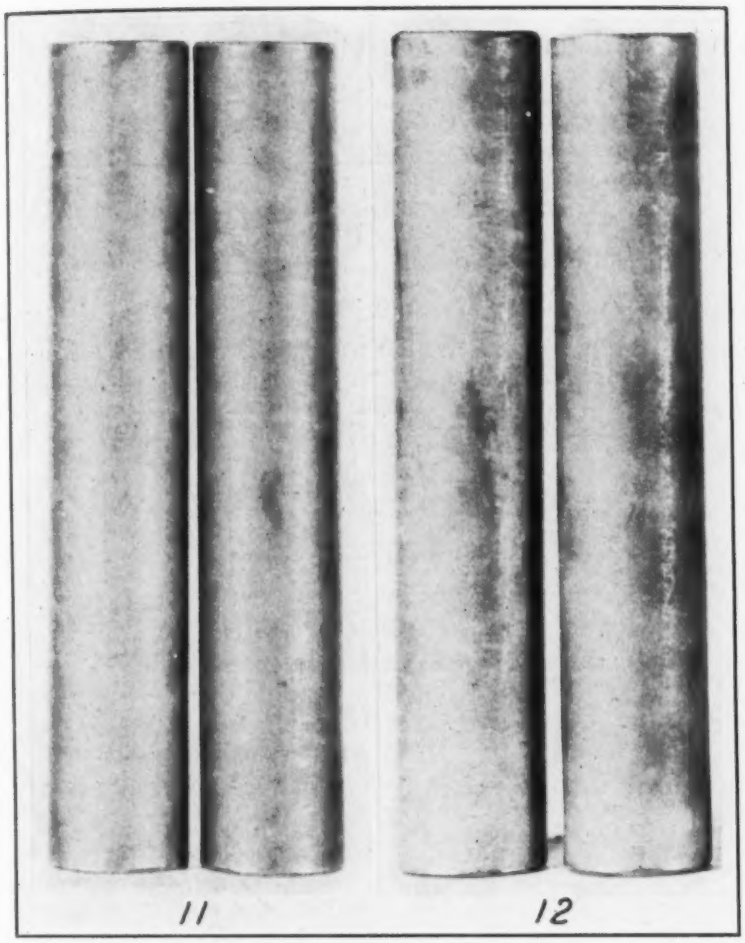


Fig. 11—Photograph of Nitralloy Specimens After Immersion in Crude Oil at 150 Degrees Fahr. for 100 Hours.
Fig. 12—Nitralloy Specimen After Immersion in Cold Crude Oil for 4 Weeks.

Hot or cold crude oil shows no attack on the nitrided tests. A slight discoloration may be noted from the hot crude oil which is not present on the cold tests (Figs. 11 and 12). The other mate-

Nitralloy—Grams Loss per Square Inch.

Crude Oil—150 degrees Fahr. for 100 Hours	Crude Oil—Cold for 4 Weeks
4 tests show no loss.	4 tests show no loss.

rials, Toncan, pure iron, copper-bearing steel, common open-hearth and Enduro KA2 likewise showed no attack.

D—5 Per Cent Sodium Hydroxide at 150 Degrees Fahr. and Cold

The hot solution was maintained at a temperature of 150 degrees

Fahr. for 100 hours. The cold test ran for four weeks, weighings made and the sodium hydroxide solution changed each week.

Nitralloy—Grams Loss per Square Inch.				
5 Per Cent NaOH—150 degrees Fahr. 100 hours			5 Per Cent NaOH—Cold 4 weeks	
Test 1—0	grams		Test 1—.00125	grams
Test 2—0	grams		Test 2—.00286	grams
Test 3—.00225	grams		Test 3—.00194	grams
Test 4—.00051	grams		Test 4—.00071	grams
Avge. Loss .00069	grams		Avge. Loss .00169	grams

The 5 per cent sodium hydroxide solution at 150 degrees Fahr. or at room temperatures shows no attack on the nitrided case. The

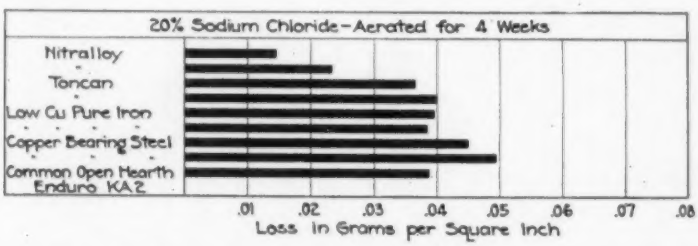


Fig. 13—Chart Showing Action of 20 Per Cent Sodium Chloride Aerated.

extremely small weight loss yields no evidence of corrosion, the test samples retaining their silver gray appearance after the hot and cold immersions.

E—20 Per Cent Sodium Chloride Still and Aerated

Both of the 20 per cent sodium chloride tests were conducted for four weeks. The solutions were renewed and weighings made each week. For the aerated testing, a flow of several pounds of air was bubbled through the sodium chloride solution.

Nitralloy—Grams Loss per Square Inch		
20 Per Cent NaCl Still		20 Per Cent NaCl Aerated
Test 1 .00787		See Fig. 13
Test 2 .00768		for data
Test 3 .00563		and comparison.
Test 4 .00624		
Avge. Loss .00685	gram	

Rust spots and pits are found on the ends and surface of the 20 per cent sodium chloride aerated tests. The pits at the surface are small but when cleaned with the point of a knife are found to be deep and extend back under the unattacked portion of the case.

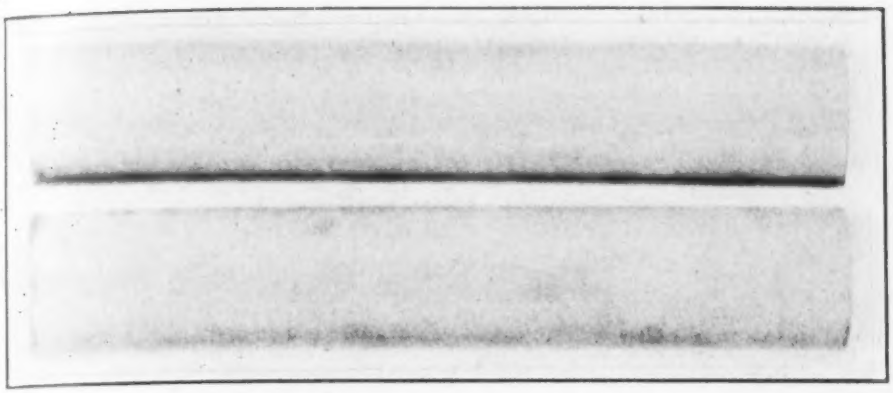


Fig. 14—Photograph of Samples Subjected to 20 Per Cent Salt Spray for 288 Hours.

The nitrided surface apparently resists this solution but once the case is penetrated the core is rapidly attacked.

The greater weight loss on tests 1 and 2 is not justified in this test for either the still or aerated solutions and may be explained. Notches were cut in the ends for identification and are deeper than the nitrided case, exposing the base metal or core. This material is severely attacked.

The 20 per cent sodium chloride solutions show no attack on the nitrided surface other than the pit attack which was greater for the aerated test than for the still test.

In the comparison chart, Fig. 13, it will be seen that the KA2 which shows no attack is the only one superior to the nitrided case.

F—20 Per Cent Salt Spray

Samples were exposed to the action of salt spray testing for four 72-hour periods—a total of 288 hours. Ten pieces were subjected to this test. None of the samples tested showed any weight loss after 288 hours.

Considering that most steels are tested for only 72 hours in the 20 per cent salt spray, Nitralloy shows a marked resistance to such an attack. No pits or any other marks of corrosion were noted on any of the samples tested (See Fig. 14). Based on these results, the salt spray test was more or less standardized as a criterion of nitrided resistance to corrosion.

When it is considered that the ordinary corrosion resistant irons and steels will rust readily under the action of the salt spray even

after 10 hours and the stainless irons must have their surface specially prepared to withstand this action, the nitrided surface shows excellent resistance.

G—3 Per Cent Sodium Chloride Solution—Alternately Wet and Dry

Nitrided samples were placed on a continuous chain which passed through a 3 per cent salt solution for about one minute and then allowed to dry in air for approximately 25 minutes. This test was run for one month or about 1500 complete cycles.

Nitralloy—Grams Loss per Square Inch
3 Per Cent NaCl—Wet and Dry
4 weeks

Test 1	.0391
Test 2	.0403
Test 3	.0397
Test 4	.0371
Average Loss	.0390 grams

The alternate wet and dry testing in the 3 per cent sodium chloride solution for four weeks showed a pit attack on the surface and ends of each sample. This corrosion was similar to that noted in the still and aerated 20 per cent sodium chloride test.

H—Weather Corrosion

To determine the corrosion resistance under atmospheric conditions, samples were placed on the test fence during the months of May and June, 1929 in the mill yard at the South Division Sheet Mill of the Republic Steel Corp., Canton, Ohio. All of the samples were stained from smoke, several showed a brown or rust tint, while others retained their silver gray nitrided appearance. All the tests were easily cleaned and showed no weight loss after one month, appearing exactly as before the test.

Additional tests were exposed to weathering for periods of 3, 6, 9 and 12 months. The latter test has not been completed. However, examination of the 9 month samples shows a slight attack on the surface. The 3 and 6-month samples showed no attack, retaining their original color.

Considering the unfavorable atmospheric conditions to which these nitrided samples were subjected, it appears evident that Nitralloy will be moderately satisfactory in weather corrosion.

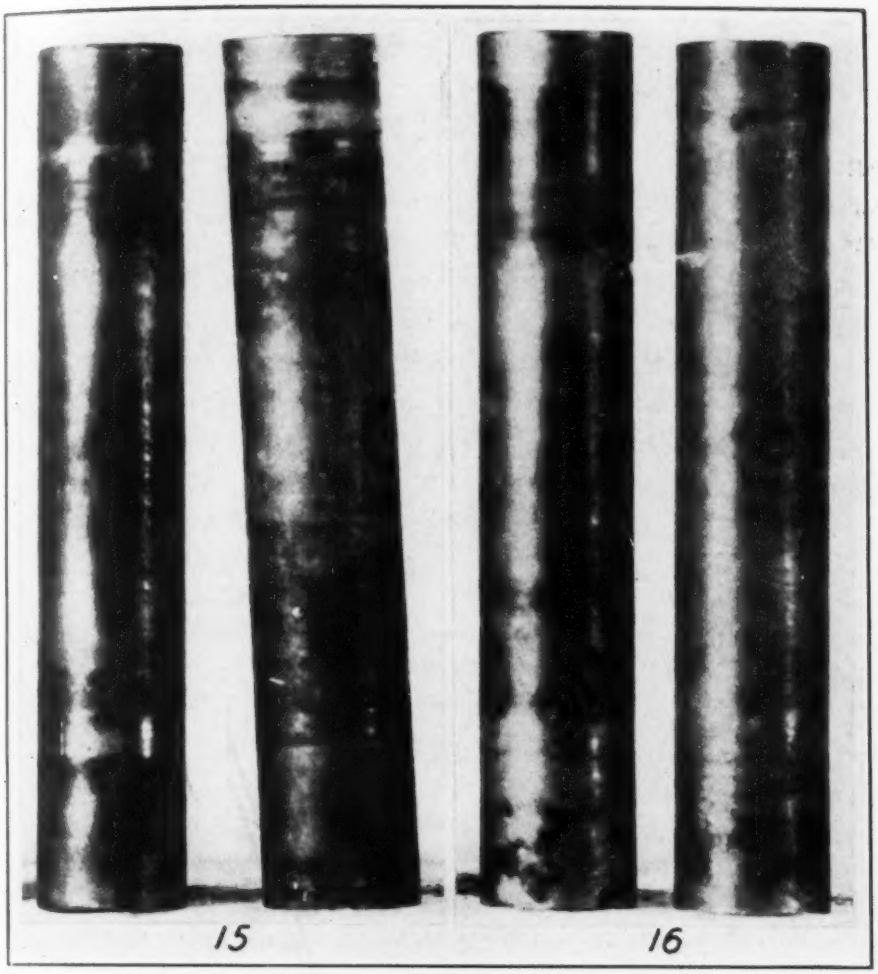


Fig. 15—Photograph of Specimens Subjected to Still Tap Water.
Fig. 16—Specimens Subjected to Aerated Tap Water. Both Sets of Specimens Revolving at Alternate Intervals in Brass Bearings.

I—Burned Natural Gas Fumes from Argand Burner

Nitrided tests were exposed to the fumes of an Argand burner using natural gas. The temperature of the fumes was maintained between 675 and 775 degrees Fahr. for a period of eight weeks. After this two-month test a discoloration of the nitrided surface showed up, which is due to a slight oxidation. No loss in weight was noted.

J—Revolving in Brass Bearings Immersed in Tap Water Still and Aerated

Brass bearings were turned so that the total clearance was ap-

proximately 0.002 inches. The nitrided samples revolved in the bearings continuously for four weeks immersed in tap water still and aerated.

A corrosion test of this nature presents the factors necessary for electrolytic corrosion. There was apparently no action of the brass bearing on the nitrided samples, no etchings, pits or other signs of corrosion were found after one month of still or aerated testing.

K—Revolving at Alternate Intervals in Brass Bearings Immersed in Tap Water—Still and Aerated

The same bearings and samples that were used in the continuous run were used for the alternate testing. The bearing and nitrided test were revolved for two hours and then allowed to remain at rest for 22 hours. This 24-hour cycle was repeated for four weeks in still and aerated tap water.

Several small pits were noted on both the still and aerated tests. In each case the pitting attack was localized to the ends of the test samples. The surface was in excellent condition and showed no evidence of pitting or corrosion (Figs. 15 and 16). The markings noted on the samples are polished areas due to abrasion of the bearing. Additional testing in bearings failed to develop pits on the ends of the nitrided tests.

L—Comparison of Stainless Irons and Nitralloy in Brass Bearings in Tap and Salt Water—Still and Aerated

Enduro Type A (0.10 C, 16.0 Cr), Type S (0.10 C, 14.0 Cr), and KA2 (0.10 C, 18.0 Cr, 8.0 Ni)—¾ inch rounds by 4 inches long with nitrided tests—0.715 inch diameter by 4 inches long—were subjected to the following tests for a comparison of corrosion resistance:

1. *Tap Water Continuous*—Two samples representative of each analysis were revolved continuously in brass bearings which were submerged in tap water for 30 days. One sample of each was in still and aerated water respectively.

2. *Tap Water Periodical*—Same as above except that the samples were revolved only 2 hours of each 24 hour day. This test run for 30 days.

3. 5 *Per Cent Salt Water Continuous*—Same as (1) except that salt water was used in place of tap water. Test run 30 days.

4. 5 *Per Cent Salt Water Periodical*—Same as (2) except that salt water replaced tap water. The test was run for 30 days.

No attack of any kind was found on the continuous and periodical, still or aerated tap water tests. The samples were turned slightly dark from the abrasive contact with the brass bearings. No pitting was noted on the ends of the Nitralloy tests as mentioned under (K).

In the continuous salt water run, some minute pits were noted on the surface of the Enduro S and KA2. These are more prominent in the periodical run, particularly so in the aerated periodical test. The Enduro S was badly pitted on the ends as was also the Enduro A. The surface of all the nitrided tests was somewhat darkened but otherwise in perfect condition. No pits on the end or on the surface were noted.

In general the Enduros and Nitralloy appear to resist any corrosion that would tend to take place when in contact with copper in tap water. The chloride content of this tap water is about 24 parts per million. When 5 per cent sodium chloride is added, the chloride content is sufficient so that, when copper is present the typical copper chloride attack takes place on the Enduros. The Nitralloy appears very resistant to the same attack.

Based on the results of these tests, Nitralloy appears to be adaptable for certain pump shaft and other applications. Many of such applications are on a production basis and are giving entirely satisfactory results.

M—Calcium Chloride

Nitrided tests were exposed to still and aerated solutions of 3 per cent calcium chloride. The tests ran for six days.

There was no loss in the still solution although specimens darkened. In the aerated solution there occurred a pitting where the specimens were stamped (the stamping of test number was done previous to nitriding). Apparently the localized cold working caused by stenciling was not removed by the nitriding temperature, and it is a well known fact that cold working of metals decreases their corrosion resistance.

N—Ethyl Gasoline

Recent work by W. J. Merten[2] mentions the failure of nitrided

[2]W. J. Merten; "Softening of Nitrided Steels by Chemical Decomposition of the Nitrides,"—Transactions, American Society for Steel Treating, Vol. 17, May 1930, p 641.

valves in internal combustion engines which use ethyl bromide compounded gasoline fuel, a pit attack being noted on the valve surface which permitted the valve to leak. Investigations of similar applications reveal that the temperatures common to such parts prohibit the use of Nitralloy. Exhaust valves may reach temperatures of 1400 to 1500 degrees Fahr. or higher. Nitralloy will not stand up for even a short period of time at temperatures above 700 to 800 degrees Fahr. without scaling.

Samples representative of various nitriding temperatures and time factors were placed in ethyl gasoline for a period of 2 months. The solution was renewed each week. An unnitrided sample was included to determine the effect of ethyl gasoline on unnitrided Nitralloy.

The sample not nitrided showed after a period of two weeks, several small centers of a corrosive attack. The various nitrided samples which were inspected weekly showed no evidence of pitting or other corrosion, after eight weeks.

Several piston pin applications have proven satisfactory, one of which is assembled in a bus motor. This has been in service over one year and is as yet satisfactory. In airplane motors, nitrided piston pins are running direct in aluminum alloy connecting rods with very promising results.

O—Salt Spray Effect of Nitriding Temperatures on Corrosion Resistance

To determine the effect of nitriding temperatures on the corrosion resistance of Nitralloy G, samples representative of seven heats were nitrided in the following cycles for salt spray testing—20 per cent—100 hours.

Run	Temperature Degrees Fahr.	Time Hours	Average Dissociation Per Cent	Flow Liters per Hour
A	950	48	35	50
B	1000	48	50	50
C	1100	48	90	50
D	1200	48	100	50
E	950	48	25	200
F	1000	48	40	200
G	1100	48	75	500
H	1200	48	90	500
I				
Duplex Run	1180	15	975 degrees Fahr.—15 Hours	
J				
Duplex Run	975	40	1180 degrees Fahr.—40 Hours	

A comparison of the samples nitrided at 950, 1000, 1100 and 1200 degrees Fahr. with flow variations suggests two stages of corrosion resistance. The high flow 200 to 500 liters per hour shows for each nitriding temperature a higher degree of resistance than for the corresponding temperature with the low flow of 50 liters per hour. A greater resistance may be noted for the 950 to 1000 degrees Fahr. temperatures than for the 1100 to 1200 degrees Fahr. nitriding temperatures.

The duplex runs I (1180-975 degrees Fahr.) and J (975-1180 degrees Fahr.) were comparative to the 1200 and 950 degrees Fahr. temperatures respectively, i.e., the high-low duplex carried the characteristics of the high nitriding temperature (1200 degrees Fahr.) and the low-high duplex (975-1180 degrees Fahr.) was characteristic of the 950 degrees Fahr. nitriding temperature.

All of the tests were approximately ½ inch square by 1 inch long, and had been ground on a 3 degree taper for gradation hardness previous to salt spray testing. This taper-ground face (case to core) greatly facilitated the above classifications; in each case the tapered face was readily attacked and showed a gradational resistance to salt spray corrosion. Pitting was not noted on any of the nitrided surfaces.

A more recent series has been completed, in which material representative of Nitralloy G—¾ inch round by 4 inches long—as annealed and as heat treated (1750 degrees Fahr. water-quench—1350 degrees Fahr. temper) was nitrided for 50 hours at 900-950-1000-1100 and 1200 degrees Fahr. with two duplex runs—1180-975 degrees Fahr. and 975-1180 degrees Fahr. for corrosion resistance.

No classifications may be made to distinguish between the annealed and heat treated material.

No pitting has been found on the surface or ends of the various tests after 100 hours of salt spray testing.

P—Corrosion Resistance of Nitrided Carbon and Alloy Steels

In one of the author's discussions of the paper presented by Harder, Gow and Wiley[3] at the Cleveland convention of 1929, gradation hardness curves were shown for the following analyses:

[3]Messrs. O. E. Harder, J. T. Gow and L. A. Willey, "Researches on Nitriding Steels"—Nitriding Symposium, 1929, pp. 141 and 142.

No.	C	Mn	Cr	Ni	Mo	V
A	.14	.30	...	...	...	...
B	.04	.12	...	2.06	...	...
D	Jalcase					
E	.12	.40	...	.49	...	...
F	.14	.50	...	1.46	...	...
G	.11	.62	...	3.26	...	...
H	.11	.38	...	4.86	...	...
I	.11	.44	.62	.65	...	...
J	.13	.46	.56	1.21	...	...
K	.12	.48	1.50	3.45	...	...
L	.13	.44	.45	2.94	...	...
M	.09	.37	1.29	3.94	...	...
N	.13	.47	.60	.56	...	...
O	.14	.44	.10	.63	.12	...
Q	.13	.50	...	1.75	.25	...
R	.11	.38	.34	...	...	...
S	.13	.38	.78	...	...	...
T	.16	.46	1.35	...	...	...
U	.12	.40	.35	...	...	.15
V	.15	.67	.92	...	...	.15
X	.11	...	...	...	...	.11

Representative samples of these various carburizing analyses were nitrided at 975 degrees Fahr. for 90 hours with a dissociation of 26 to 30 per cent. It was evident that the carbon, nickel and nickel-molybdenum steels showed only a slight ability to nitride, whereas the chromium, chromium-vanadium, and the chromium-nickel analyses show a marked ability to nitride developing maximum hardness values of 600 Vickers Brinell. Duplicate tests of this series were subjected to 100 hours of salt spray testing, being inspected every 25 hour period.

None of the carburizing grades showed any evidence of attack after the first two periods, and even after 100 hours showed no pitting. It was apparent that the carbon, nickel and nickel-molybdenum steels, which developed no appreciable hardness, showed a corrosion resistance comparable to the chromium, chromium-nickel and chromium-vanadium steels which developed a hardness of 400-600 Vickers Brinell.

Q—Conclusions

From the foregoing results, it is evident that Nitralloy is not adapted for use in acid solutions, such as sulphuric or hydrochloric. It is extremely resistant, however, to alkali, atmosphere, crude oil, ethyl gasoline, natural gas combustion products, tap water and still salt water. While it is slightly attacked in aerated and alternately wet and dry salt water, there also seems to be no action between it and brass in contact either still or moving immersed in hard water.

Section III

Denitriding of the nitrided case presents an interesting and

Table I

As Nitrided Vickers	As Denitrided Vickers			As Renitrided 975°F.	
Test No.	Brinell	50-50 NaCl-KCl		Brinell*	Vickers Brinell*
1	920	1400 degrees Fahr.	30 min.	572-599	446-493
2	920	1400 degrees Fahr.	30 min.	572-599	446-514
3	920	1400 degrees Fahr.	30 min.	613-627	525-548
4	920	1500 degrees Fahr.	30 min.	460-473	446-464
5	920	1500 degrees Fahr.	30 min.	483-493	483-514
6	920	1500 degrees Fahr.	30 min.	483-493	483-536
7	920	1500 degrees Fahr.	1 hr.	483-493	493-503
8	920	1500 degrees Fahr.	2 hrs.	460-483	536-548

*These values are the low and high values of five readings.

fertile field for investigation. Applications are known and others undoubtedly will develop where after the part has been nitrided and in service, a change in design becomes necessary, which requires additional machining. Therefore to be able to denitride and soften the part, do the necessary machining operations and renitride to obtain high surface hardness, etc., would be indeed a welcome procedure. Such would well apply for example to core dies in the die casting industry and to forging die inserts and upsetting die inserts. While the denitriding and softening for machining can be readily accomplished, the renitriding presents serious difficulties in that the denitrided but unmachined parts cannot have their surface hardness materially raised by renitriding. In other words, if a nitrided case is denitrided and its hardness falls to 250 Brinell for example, the renitriding of this sample will not materially raise this hardness value. Discussion will follow later in this paper.

Methods of Denitriding

W. J. Merten[2] has proposed a method for the denitriding of ni-

Table II

Test No.	Temperature	Normalized Hardness Time	Vickers Brinell	Case Depth	Scale Thickness
1	1600 degrees Fahr.	2 hours	483-514	.025-.026"	.004"
2	1600 degrees Fahr.	4 hours	437-437	.025-.025"	.004"
3	1600 degrees Fahr.	6 hours	413-429	.021-.021"	.007"
4	1600 degrees Fahr.	8 hours	413-421	.020-.021"	.008"
5	1800 degrees Fahr.	2 hours	317-317	.019-.020"	.014"
6	1800 degrees Fahr.	4 hours	292-297	.018-.019"	.015"
7	1800 degrees Fahr.	6 hours	266-270	.017-.018"	.017"
8	1800 degrees Fahr.	8 hours	209-221	.016-.018"	.018"

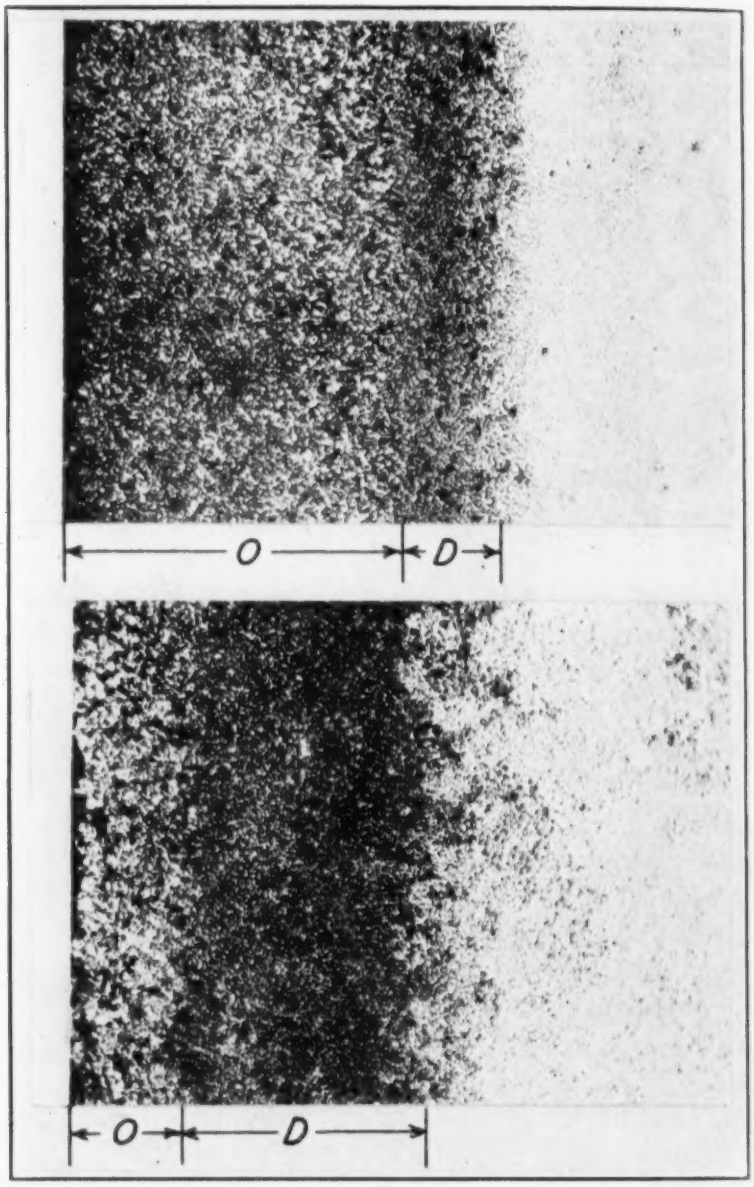


Fig. 17—(Top) Photomicrograph of Nitrided Case After Heating to 1600 Degrees Fahr. for 8 Hours. O—Remainder of Original Case .017 Inches. D—Diffused Case .003 Inches.

Fig. 18—(Bottom) Photomicrograph of Nitrided Case After Heating to 1800 Degrees Fahr. for 8 Hours. O—Remainder of Original Case .006 Inches. D—Diffused Case .010 Inches.

trided parts. Briefly summarized, his method consists of heating the part to be denitrided in a bath of sodium-potassium chloride (50 per cent NaCl-50 per cent KCl) at 1450-1500 degrees Fahr. and cooling slowly to a temperature slightly above the freezing point of

Table III

No.	As Nitrided Carbon	Nitrogen	Medium	Temp.	Time	As Reheated Carbon*	Nitrogen
A	.345	8.5	Salt	1500°F.	1 hour	.135*	.695
B	.345	8.5	Salt	1500°F.	3 hours	.105*	.593
C	.345	8.5	Iron Filings	1500°F.	1 hour	.054	.560
D	.345	8.5	Iron Filings	1500°F.	3 hours	.057	.224
E	.345	8.5	Vacuum	1700°F.	2 hours	.265	.224

*Unnitrided or green drillings subjected to same treatment show no carbon loss.

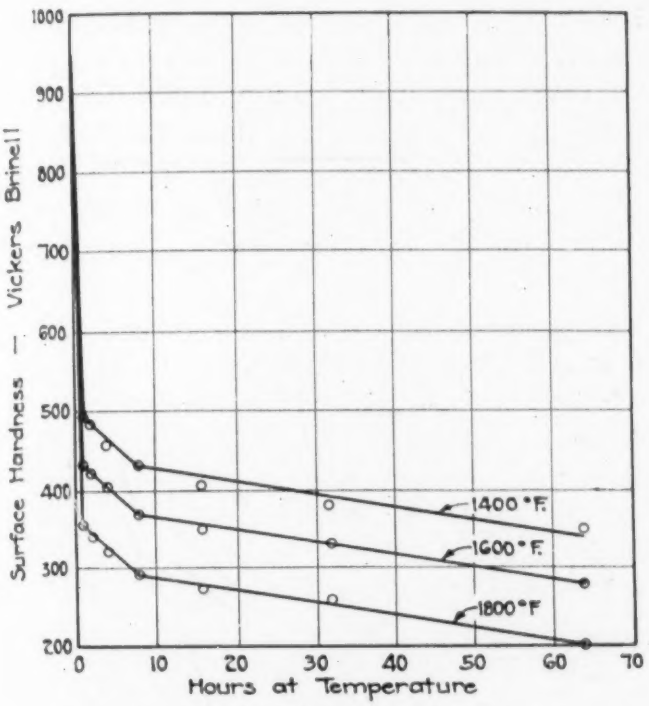


Fig. 19—Curves Showing Effect of Time and Temperature on Surface Hardness of Nitrided Case.

the fused salt. This lowers the surface hardness to 250 Brinell or lower. This loss in hardness is due to loss in nitrogen which is removed by chemical decomposition as NCl_3 nitrogen trichloride. Renitriding is claimed not to introduce any difficulties or develop any new characteristics.

Upon checking this method, the above claims are not all substantiated. Results are given in previous Table I. Specimens which had been nitrided to 920 Vickers Brinell surface hardness were denitrided, then renitrided at 975 degrees Fahr. for 30 hours. Samples nitrided for the first time in this renitriding run developed a surface hardness of 900 Vickers Brinell. From the data, it will be seen that

the hardness lost by denitriding cannot be regained by subsequent renitriding.

Prior to Merten's paper, the authors conducted a short denitriding series to determine the machining possibilities after normalizing from temperatures of 1600 and 1800 degrees Fahr. with renitriding in view. Several nitrided specimens (½ inch square by 1 inch long) having a Vickers Brinell of 946 to 1003 and a case depth of 0.025 inch were reheated at 1600 and 1800 degrees Fahr. for periods of 2, 4, 6 and 8 hours. Table II shows the resulting hardness values, case depths and approximate scaling values. A comparison of the case depths and scale thickness (or decrease in cross-section) with the original case depth clearly shows that the case actually is migrating or diffusing towards the center. In Figs. 17 and 18 are shown photomicrographs of the effect of 8-hour periods at 1600 and 1800 degrees Fahr. The section of the case marked "O" represents that portion which remains of the original case while the area of case marked D represents that portion of the case which has diffused towards the center. Thus at 1600 degrees Fahr. for 8 hours the case has diffused inward 0.003-0.004 inch while at 1800 degrees Fahr. for 8 hours this value has increased to 0.010-0.012 inch.

This normalizing method when conducted from 1800 degrees Fahr. allowed the case to be readily machined as will be inferred from the hardness values. However, it was necessary to remove completely all of the original and diffused case before satisfactory renitriding could be accomplished.

In an attempt to determine the necessity of the double chloride bath at 1400 and 1500 degrees Fahr. nitrided samples were packed under iron filings, to prevent surface oxidation, and subjected to temperatures of 1400, 1600 and 1800 degrees Fahr. for periods of 1 to 64 hours at respective temperatures. Specimens were cooled to room temperature under the iron filings. The results of this test indicate that the hardness loss is due to temperature and time and not dependent upon heating media. These results are shown in chart form in Fig. 19.

A further attempt to prove that the softening of the nitrided case is due to a function of time and temperature and not media was made by reheating of nitrided drillings in the double chloride salt bath, under iron filings and in vacuum. The nitrogen and carbon content of the drillings were obtained as nitrided and after the various methods of reheating. Table III shows the results of these

tests. It is again evident that time and temperature are the prime factors for accomplishing the denitriding.

Conclusions

A review of the preceding work on denitriding clearly points to the following facts:

1. The denitriding is dependent upon the temperature and time and not the media.

2. The lower hardness of a denitrided case is the result of an appreciable loss of nitrogen content.

3. Denitriding at temperatures above 1400 degrees Fahr. is accompanied by a marked diffusion of case toward the center. The rate of diffusion is directly proportional to temperature.

4. In order to obtain high or maximum surface hardness by subsequent nitriding, it is necessary to remove entirely all of the denitrided case.

DISCUSSION

Written Discussion: By J. H. Higgins, material engineer, Camden Forge Co., Camden, N. J.

The paper presented by Messrs. Sergeson and Deal is very interesting as well as instructive. I am especially interested in the description of their enameling experience.

With the thought of prolonging the life of our structural steel container, we attempted to enamel the inner surface, but with very sad results. The surface was first thoroughly sand blasted, then sprayed with a hard enamel and baked at 1550 degrees Fahr. The result was a mass of thin bubbles which brushed off with the sweep of a hand. This condition was entirely removed by repeated sand blasting; the box was filled to the brim with a 10 per cent solution of sulphuric acid and left standing for 24 hours. This formed a pronounced scale which was again removed by sand blasting, then sprayed and again baked at 1550 degrees Fahr. After this second baking the surface showed a mass of globules which fell off easily when bumped; at no place in the box did the enamel stick as a uniform layer. I am of the opinion that the nitrides present on the surface of the box prevent, by some chemical reaction, the adhesion of enamel. In this respect I would ask Mr. Sergesen if any of his experiments were conducted on materials which had been previously subjected to ammonia gas. Further enameling experiments are being made at our works on both iron and steel, but on new stock.

The proper seal for large containers is of prime importance. Rose metal, due to its low melting point, is considered one of the best. This material is quite expensive, which probably prohibits its general use.

We are using lead, and while the seal is satisfactory the melting point is

rather high, which in some respects is objectionable as the steel absorbs too much heat before the lid drops in place.

W. J. MERTEN[1]: The part of Mr. Sergeson's paper that I want to discuss is the de-nitriding feature. When the paper came to me I was surprised to find that the de-nitriding of parts which are highly and accurately finished could possibly be undertaken by anyone at 1800 degrees Fahr. for softening for alteration. This temperature naturally involves decarburization and warping and would probably scrap the material because the highly finished fitting parts are unfit after reheating, even in a vacuum warping would be so intensive that one could not use the material again. I also want to call attention to the fact that when I gave my paper on de-nitriding in a salt bath I particularly stressed the fact that one of the materials which I subjected to de-nitriding was a strip of $\frac{1}{32}$" thickness, nitrided clear through the cross section, so diffusion was out of the question as far as the softening was concerned. The same strip heated to normalizing temperature would scale all right, but it didn't lose any hardness. I also want to inform Mr. Sergeson that we are doing de-nitriding practically every other day successfully and without the detrimental effects that he has obtained in his practice. I would like to get some more details as to the procedure which he employed in order to get the results he describes.

J. P. WALSTED[2]: The question of nitriding containers has been the most troublesome that has been encountered in the development of the nitriding process. Of the containers used, nickel and monel metal have been the most satisfactory. One manufacturer of nitriding furnaces after conducting many tests over a long period has selected monel as the best material.

Iron and low carbon steel is eliminated for obvious reasons which Mr. Sergeson and Mr. Deal have mentioned in their paper. The high chromium, low carbon stainless iron has been shown to be unsatisfactory.

The containers having an indefinite life, such as is the case with those made of monel metal or enamelled steel, the problem has been solved. However, there are some things that need explaining. For instance, we had at M. I. T., two furnaces, one fitted with a pure nickel tube, the other fitted with a chrome iron tube. The chrome iron tube ceased to function after several hundred hours of service. The nickel tube was giving perfect service after more than two years of constant use.

Information was received from a reliable source that heating to 1650 degrees Fahr. for several hours would cause the chrome iron tube to recover. This was done with some success. In order to forestall trouble with the nickel tube, it was given the same treatment. No satisfactory results have been obtained from the nickel tube since that time. Sand blasting failed to eliminate the difficulty. Leaching with alkalis and acids was of no value. The tube is still in the laboratory, apparently as good as new but actually worse than useless.

An 18-8 tube gave good results for several hundred hours, then suddenly

[1]Metallurgical engineer, Westinghouse Electric and Mfg. Co., East Pittsburgh.

[2]Instructor in physical metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

ceased to function. Leaching with a three acid mix caused complete recovery, but the tube lasted for a very short time.

Samples placed in an 18-8 tube and nitrided for 48 hours were nitrided perfectly on one side, the remainder being untouched. Occasionally a sample 4½ inches long would be nitrided for two-thirds its length, the remainder remaining soft and bright. The unnitrided areas had no relation to direction of ammonia flow nor to position in the tray. Samples placed in a furnace and nitrided for 48 hours came out without a trace of nitriding. Replaced in the furnace for 48 hours with conditions identical, these same samples came out perfectly nitrided.

I give these experiences to show that there are many unexplained factors in this simple process of surface hardening. Another reason is to correct the impression that nitriding is valueless. Poor nitriding has been responsible for the rejection of the process for many applications where it is entirely suitable. This is unfortunate, but will be corrected by proper nitriding of parts in satisfactory containers.

The resistance to corrosion after nitriding shown by steels that do not harden by the ammonia treatment is not new. It was first noted by Despretz in 1829. There can be no doubt that nitriding of pure irons and carbon steels will protect these materials against corrosion. When the brittleness caused by the nitride needles is not of great importance the use of the nitriding process as a corrosion protection is entirely possible.

A. B. KINZEL[3]: There are two phases of the paper that I should like to discuss. One is the matter of containers. In our work, particularly in the early work, we had much trouble duplicating results because of containers. We even used a new container each time. Since then we have learned to use the insert materials and we have had absolutely no trouble with uniformity whatsoever. It is possible that this insert material scheme will eliminate the container problem and that of course is tied up with the economics of a large unit.

The other point is the matter of corrosion resistant steels. That Mr. Sergeson got the salt spray results reported is true without question. I happen to have seen them. Unfortunately however we have not been able to get these results ourselves at the laboratory. That may be because we use a different type of corrosion testing. Both tests are called salt spray tests but the set-up is different. Perhaps that is the answer, or the answer may be in the nitriding. The point to be emphasized in this connection is that brought up by Mr. Walstead, namely, that the resistance to certain corrosion is increased by nitriding in all steels but to a different degree in different steels.

D. LEWIS[4]: Mr. Sergeson is very much to be congratulated on a paper which is of vital interest to everybody doing commercial nitriding. With reference to the deterioration of the box I do not think that is properly understood. I do not see any reason why nitriding should be any different if your dissociation remains the same. In other words if you have to increase the ammonia

[3]Associated with Union Carbide and Carbon Research Laboratory, Long Island City, N. Y.

[4]Research engineer, American Chain Co., Bridgeport, Conn.

flow to 500 per cent, I do not see why that should make any difference providing you have the same composition of gas coming off from the box. I will confine my discussion to one other point, the matter of corrosion resistance. We find that corrosion resistance is confined to a very thin film at the surface, and that if as much as one-thousandth of an inch of surface material is removed most of the corrosion resistance disappears. That is particularly well shown by taking a taper ground piece used for determining the hardness through the nitrided case and exposing it to atmospheric corrosion. It will corrode practically all the way up the taper to the surface. Under the microscope we find that there is at the surface a white etching layer. Mr. Sergeson has mentioned that in an earlier paper. That layer is about the same order of thickness as the corrosion resisting film. We wonder whether the corrosion resistance is due to that white etching layer. We would be very much interested to know whether anybody has tackled the corrosion resistant properties from that angle.

Authors' Closure

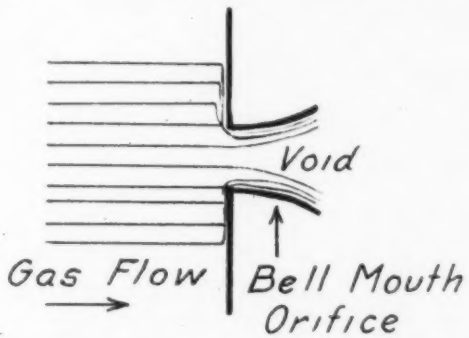
Mr. Higgins mentioned the fact that he tried without success to enamel a container that had already been used for nitriding. The fact is that when we bake enamel on a container we have to reach a temperature of 1450 degrees Fahr. or higher. In doing this the nitrogen, previously adsorbed by the walls during nitriding, is driven out and pushes the enamel from the surface. It is not practical to enamel the surface unless we can in some way remove the nitrogen from the container walls.

Mr. Merten calls attention to the fact that when we reheat to 1800 degrees Fahr. we distort the piece and make it worthless as far as re-nitriding is concerned. That is correct, but the fact remains that we cannot soften the piece unless we heat it to that temperature (1800 degrees Fahr.). The lower temperature (1450 degrees Fahr.) decreases the hardness to approximately 450 Brinell. Subsequent nitriding will not materially raise this hardness; this has been proven. Last year we ran some experiments which I think will bring out the point. We nitrided square pieces at 950-1000-1100 and 1200 degrees Fahr. These were ground on a three degree taper and hardness values obtained down the taper. The freshly ground tapered surface was then renitrided at 950 degrees Fahr. It was expected that all the tapered surfaces would show the same hardness throughout, that is all would be 1000 Vickers Brinell. Such however was not the case with the exception of the 950 degree Fahr. run and to some extent the 1000 degree Fahr. run. The 1150 and 1200 degree Fahr. runs show the hardness curve to follow the original curve until the core is reached, when the hardness jumps up to a high value (illustrates on board). These curves are to be found on page 173 of the Nitriding Symposium—A. S. S. T. 1929. I believe this shows conclusively that once we soften the case by de-nitriding we cannot reharden the piece to its original high hardness, i. e., around 1000 Vickers Brinell. We tried to follow out Mr. Merten's details in heating our specimens in a 50-50 double chloride, of sodium and potassium, salt bath for various lengths of time and cooling in the salt mixture to near its melting point before removing specimens to air. The melting point of this mixture is around 1220 degrees Fahr.

In regards to the subjects mentioned by Mr. Walstead, we have used

nickel tubes and found them to become brittle. In some cases they have cracked very early which may have been due to the so-called season-cracking due to cold work in tubes. The nickel tubes were cold drawn. We also have used high chrome tubes (0.10 per cent carbon 16.0 per cent chromium) and have obtained fair results. At present we are using ordinary carbon steel tubes and replace them when they become brittle or break. We have never tried pickling solutions for reviving tubes so I cannot reply to that question.

I was at a lecture the evening before last and heard something which may explain the irregularity which Mr. Walstead has found, i. e., soft spots on



nitrided specimens. I imagine he is referring to nitriding in small tubes. This lecture was a study of the flow of gases. These gases were photographed at the rate of 40,500 photographs a second. It was interesting to note that the gases flowed in a straight line until they hit some object. As the gases left different orifices they took definite courses. In Fig. 1 is shown the flow of gas from a bell mouth, note the apparent void. Were the gas a nitriding gas and specimen placed in the void, a soft surface might be expected. A study of the flow of gases might be a very interesting one to learn why we get such strange results in some of our nitriding for which, for some reason or other, we find no explanation.

Mr. Kinzel mentions containers and has used an iron container. The containers do not fail due to breaking. It is due to the fact that we do not get satisfactory results out of the container that we have to scrap it. Apparently if Mr. Kinzel uses his packing material and introduces the ammonia into the packing material, the ammonia reaches the work before it reaches the sides of the container. Mr. Kinzel also mentions erratic results in the salt spray. We have found by placing drillings in our runs and checking the nitrogen content of drillings after the run, we do not have full corrosion resistance if a low nitrogen (approximately 4 per cent or less) content is obtained. Apparently the corrosion resistance is dependent upon the nitrogen content of the surface.

Mr. Lewis has asked if we maintain 20 per cent dissociation regardless of rate of flow, why should we not obtain the same results. That we do not obtain the same results can be ascribed to the condition of the nitriding container. The longer the container is used the more it breaks up the ammonia on the walls, and after all we are determining the dissociation by means of the exit gases. How much ammonia breaks up upon the walls and how much breaks up upon the work are the determining factors.

NITRIDING FURNACES AND EQUIPMENT

By W. J. Merten

Abstract

This paper describes the furnace equipment of a nitriding plant in which a practically continuous nitriding process is successfully conducted. The equipment consists of two pairs of stationary twin containers heated by two removable bell type electric furnaces. One bell furnace serving alternately two containers thereby approaching continuity of operations as well as introducing economical features of great importance. Consistently uniform and selective quality of product are assured by an equipment permitting a practical reproduction of conditions essential for obtaining such results.

Introduction

THE ever increasing interest in nitrided steels for parts requiring unchanged physical dimensions after hardening, along with high hardness, extreme wear resistance, combined with retention of hardness at elevated temperatures and corrosion resisting properties has resulted in a tonnage demand for nitrided parts which compels a careful study of the nitriding equipment for practical, economical and efficient processing.

Nitriding while apparently a simple process is quite sensitive as to details of procedure and unless careful attention is paid to these details, unsatisfactory results are obtained.

Prime Factors for Successful Nitriding

The most essential points to be observed in nitriding are:

1. Constant temperature control for the entire time period of the process
2. Uniform distribution of active nitriding gases
3. Ejection of hydrogen gas H_2, the product of decomposition of ammonia, detrimentally affecting nitriding

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, W. J. Merten, a member of the society, is metallurgical engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh. Manuscript received June 13, 1930.

4. Gas sealed container to prevent leaching of oxygen from the outside atmosphere

5. Inert container and pipe material on all parts subjected to hot NH_3 gases.

Item 1 obviously involves principally the furnace proper. The low temperature 1000 degrees Fahr. at which nitriding is conducted pre-eminently fits the metallic resistor electric furnace for this work.

The furnaces described are the outcome of an intensive study of the work that has been done by others as well as the experience at our own plant and an attempt has been made to eliminate most of the objectionable features of the various designs and installations.

Furnace Equipment

Fig. 1 shows the general plan of the installation. A, B and C, D are pairs of stationary containers mounted on refractory pedestals. C, D is the larger pair, the internal dimensions of the boxes are 36 inches wide, 75 inches long, 28 inches high. A, B is the smaller pair of containers, of 28 by 36 by 18 inches internal dimensions. The pedestals carry heating elements on top for heating direct through the bottom of the container.

B and C show the two bell type furnace housings in place. One bell housing for a set of two boxes provides for a very interesting and practical process of nitriding, a detailed description of which follows.

The two outstanding characteristics of this arrangement are: First: Stationary boxes or containers and consequently stationary or fixed ammonia gas piping, the importance of which cannot be overemphasized for quality work, in view of the fact that leakage of joints will always cause leaching of air into the container and produce inferior results. Oxide films do retard nitriding materially and also prevent uniform formation and diffusion of the nitrides. Pushing and pulling the containers in and out of the furnace even on conveyors subjects the containers to straining, affecting the useful life of the box, making necessary frequent repairs especially on welded joints. Obviously, welded joints are unavoidable in large equipment for economical reasons. Moving of the box and the network of piping introduces heavy straining of joints and distortion, accompanied by cracks and consequent leaking. Probably more unsatisfactory nitriding is due to nonsealing joints in ammonia piping and

open welded seams of boxes and poor cover seals than to any other factors influencing the quality of the nitrided product. Perfect seals also have a decided influence upon the life of the equipment since repairing of containers usually involves local heating to high temperature if such repairs require rewelding as is ordinarily the case. Again, the presence of unsatisfactory seals is usually detected after the completion of the process from the appearance of the nitrided parts, or when the injury due to such leaks is done. The decomposi-

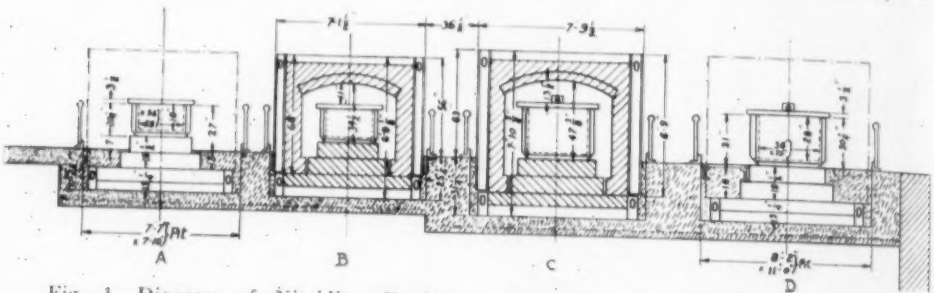


Fig. 1—Diagram of Nitriding Equipment Layout Showing Bell Type Furnace and Stationary Twin Containers.

tion of ammonia and the burning of gases in the furnace heating chamber for the period of the process, of course, does not favor the preservation of the heating equipment.

Second: Continuity of the process operations or a long time period process, without resorting to long tunnel type furnace equipment and mechanical conveyor type of transports characterizes this equipment.

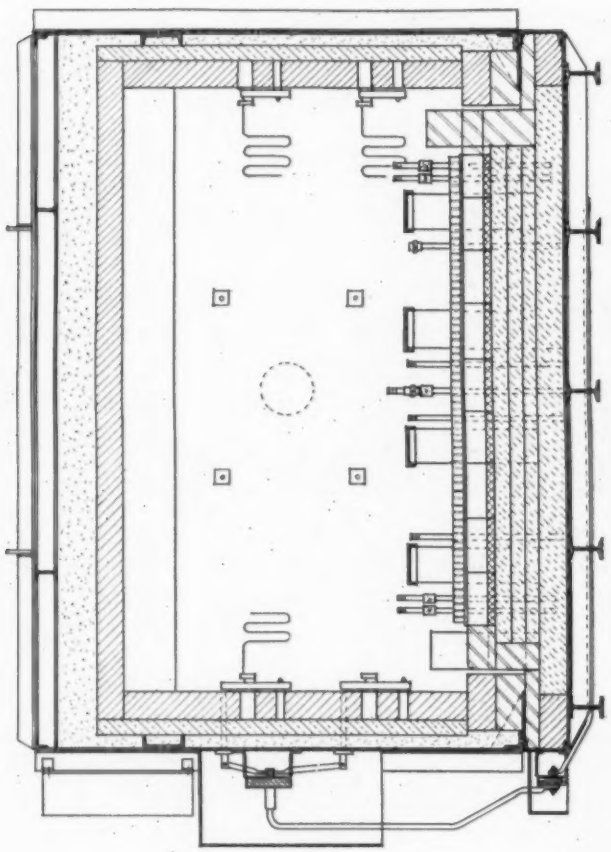
A description of the process will best illustrate this claim of continuity of performance. For starting, the first container is loaded and cover put in position and bolted or clamped as desired or provided for. The ring lead-seal heater is then turned on to heat preliminary to pouring the lead seal. The lead seal is then poured and test for leaks made and leaks eliminated. The bell furnace is then put into position and the electric current turned on, simultaneously with this starting of the heating, ammonia gas is turned on and the flow regulated just short of disturbing the lead seal. This excess flow of gas in the beginning is necessary to expel the air from the container and prevent superficial oxidation or cooling of steel parts prior to coming to heat (temperature). The flow is retarded somewhat when the temperature rises and is gradually regulated to a standard predetermined volume for the charge. Temperature control mechanism and occasional observation of gas flow through the water

seal at the end of the exit tube during and for the time period of the process will take care of the work. While this furnace is in operation the other container is charged with its load, sealed, tested and prepared for its cycle of heating for nitriding. As soon as the first container has completed the time period of processing which is 20 hours at a temperature of 975 degrees Fahr., the electric current is turned off.

The bell furnace is removed and placed over the second box prepared to receive it. The heat stored in the walls of the bell, of course, is preserved and utilized for heating the second container, shortening thereby the time period for heating up of container to the processing heat. The cooling of the first container with its nitrided charge proceeds with the ammonia gas flowing until the charge cools down to a temperature of approximately 350 degrees Fahr. The ammonia gas is then turned off and the box cover removed. The temperature now is below that at which discoloration and deformation due to sudden or nonuniform cooling occurs and will therefore not result in deleterious effects on the nitrided parts or the equipment. After unloading and recharging, sealing and testing, this box is again ready for firing. A process heating cycle of approximately 24 hours obviously is practically a continuous one, and certainly can be made so, as well as economical, with a double unit pair of containers of suitable dimensions with bell type electric heating furnaces.

Bell Type Nitriding Furnaces 50 and 75 Kilowatts

Fig. 2 is a diagram of the 75-kilowatt bell heater. It is a rectangular open-bottom, brick chamber provided with electric heating elements of nickel-chromium flat ribbon. Figs. 3 and 4 illustrate the mounting and assembly of the heating elements in the furnace chamber. The simplicity of construction is at once evident and permits a rugged economical design assuring low maintenance cost. The low temperature used in the process allows considerable handling and rough usage without injury to the furnace since the strength of the materials used in the construction is quite high and is unimpaired by the degree of heat to which it is subjected and at which handling and transporting is done. The degree of uniformity of heat transfer to the containers is amplified by additional resistor elements or heaters under the top plate of the pedestal, which is, of course, of considerable advantage in the uniform transmission of



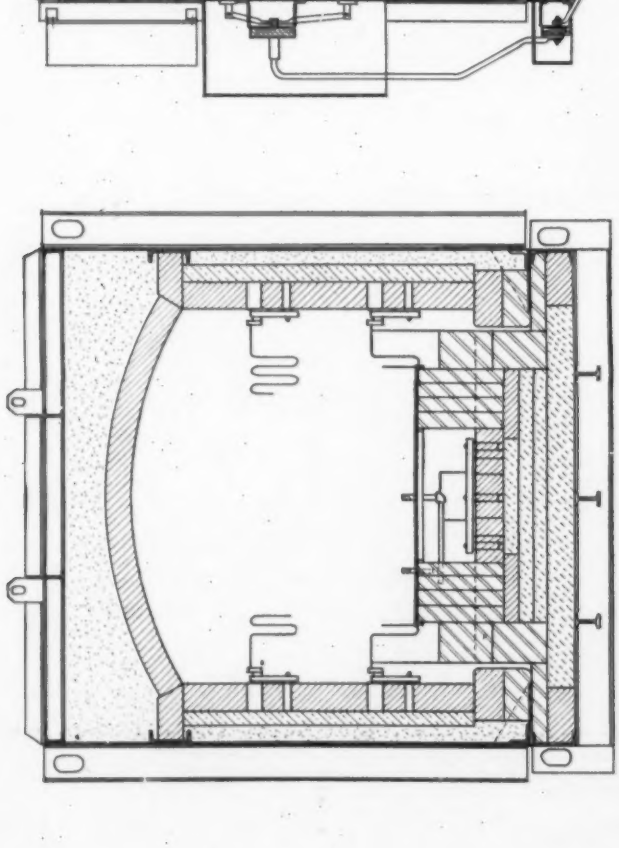


Fig. 2—Diagram of Bell Type Furnace, 75 Kilowatt, 220 Volt, 3-Phase.

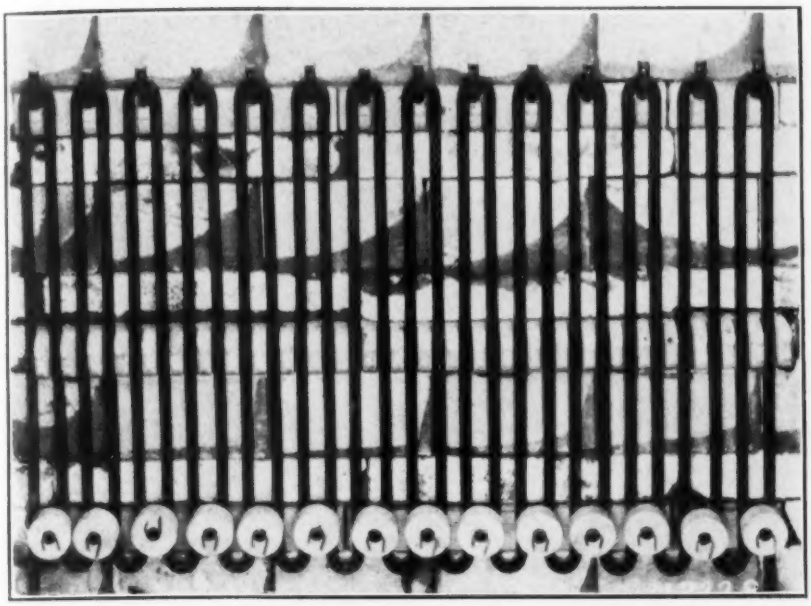


Fig. 3—Photograph Showing Mounting of Heating Elements in Furnace Chamber.

heat to the charge when properly distributed. The direct heating through the bottom of the box has certain distinct advantages which should be recognized and appreciated. In the beginning of the heating period, it aids in the ejection of air and consequently assures a clean untarnished surface of the load or charge all through the processing period.

During the active processing period it helps to prevent packing of undissolved ammonia and results in increased nitriding reaction due to the more positive upward directed movement of gases. It also provides a convenient means for the heating of the box for sealing with low temperature melting point metals, such as tin, lead, etc., at the time of placing the box cover and before placing the bell furnace.

Nitriding Containers

The design of the nitriding container (Fig. 5) involving construction of box-size and distribution of ammonia inlet and exit piping, as well as the selection of the most suitable metal for this is probably of greater importance than is generally admitted. The container sizes, of course, are determined entirely by the character or general physical dimensions of the parts to be nitrided and the quan-



Fig. 4—Photograph Showing Assembly of Heating Elements in Furnace Chamber.

tity production requirements. A combination of sets of large and small units will, in most cases, be the more practical and economical arrangement, especially in a jobbing plant and a nitriding department in a large electrical machinery manufacturing plant certainly must be considered from a jobbing angle.

Container Material

Nickel is probably the best inert metal for containers and is exclusively used for shelving, supporting members, interior ammonia piping and cover seating surfaces. Nickel is practically inert to the action of hot ammonia and the gases of dissociation, H_2 and N_2. The smaller set of boxes is made of nickel so that the entire assembled container presents an inert metal to the reacting gases during nitriding. Experiments conducted in an all nickel box have shown superiority of results both from a viewpoint of uniformity as well as higher degree of hardness. Obviously the life of such a box is prolonged considerably.

Chemical attack of hot gases upon ordinary low carbon steel plate while slow, nevertheless, is finally telling in its effect by the oc-

currence of leaks through welded seams and embrittlement of the steel. Readily applied protective coatings of inert character other than electroplated and otherwise deposited nickel for which a search is now being made, however, may change this situation entirely in favor of low carbon steel for box material and for supports, shelving and sealing surfaces. For piping, however, there will probably never be anything better nor more practical than nickel since the uniform and uninterrupted, predetermined flow of nitriding gas must be maintained and is assured by a nonchangeable inert metal. Any growth or change in dimensions of perforation of piping for gas discharge into the reaction chamber which occurs when steel or iron tubing is used will impair final results.

The box material in the large size containers is ⅜-inch nickel plate welded at edges and corners and with lead sealing channels welded on. The smaller boxes are ¼-inch nickel plate also built up by welding. Better security against leaks in the welded seams or joints would be possible and can be obtained by press forming the box part of the containers. A standardized equipment would make this economical and practical and radical changes in methods of container manufacture may be looked for in the near future.

Ammonia Supply Tanks and Ammonia Inlet Piping

A continuous flow of ammonia gas during the process is best assured by a battery of ammonia tanks connecting into a manifold from which a takeoff main supply tube line leads to the nitriding container. The tank battery should be housed as in this installation in a shed provided with heating and ventilating apparatus to keep a fairly even temperature surrounding the gas tank, through the year. A ¾-inch gas main has been found ample for a proper supply of gas for the work. The box inlet pipe is a ½-inch nickel tube having an ammonia gas regulating valve inserted between the main and the inlet pipe.

The distribution of the piping within the container is of special design and of interest since upon its correct positioning and design largely depends the uniformity of final results over the entire charge. This is, of course, of greater significance in the large size boxes than in the smaller containers.

Fig. 5 gives an illustration of the gas distributors within the container. There are three separately supplied distributors, two located at the bottom ends and one in the middle at the bottom of the

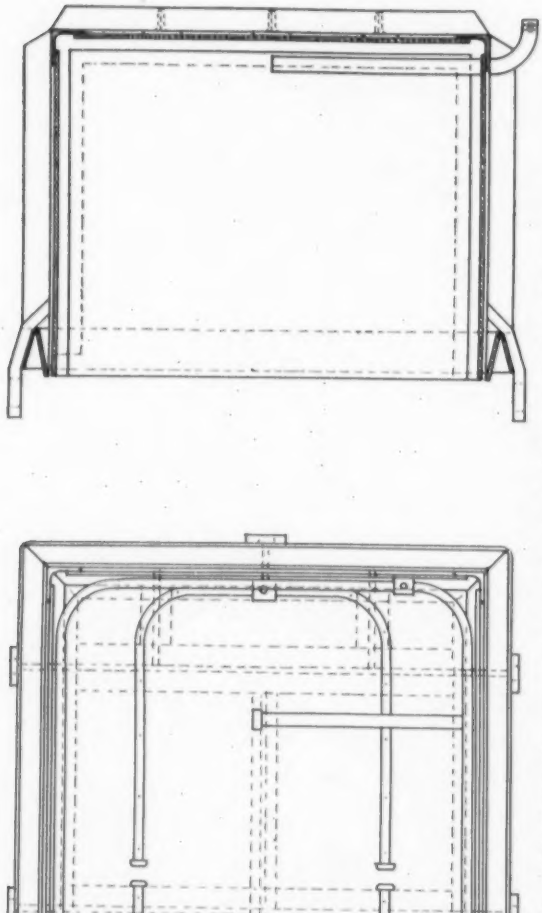


Fig. 5—Diagram of Nitriding Box for Bell Type Furnace Showing Ammonia Inlets and Exit Piping.

box. The front and rear distributors are U shaped, open towards the middle of the box and the middle one is H shaped. The ends are closed and small holes equally spaced in an obliquely upward direction are provided for gas outlets. The combined areas of the holes are slightly less than the ½-inch pipe opening, thereby forcing the distribution of the gas over the length of both arms of the U and the arms of the H.

Gas Exit Piping

The exit or take off piping in an assembly of this type must naturally be located in or near the top and should preferably have its opening in the cover over the gas distributors and, consequently, the number of openings are three in this equipment. The idea of churning or agitating the gases by fan action within the container has been definitely discouraged. We felt that since the efforts should be directed towards an ejection or elimination of the inactive gases of decomposition of ammonia, namely, hydrogen, rather than its more intimate mixing with the undecomposed NH_3 and the nascent or chemically active nitrogen. The discharge of the waste gases through a water seal for the absorption of free ammonia is retained in this plant. A slowly running light flow of water is maintained all through the process to supply a fresh absorbent for the undecomposed ammonia, reducing thereby the ammonia contents of the atmosphere surrounding the nitriding furnaces.

Liquid Lead Seal

As stated earlier in this paper, a perfect gas tight sealing of the container is the most essential factor for good nitriding results. A liquid lead sealing bath in a V grooved channel around the open top of the container into which a V shaped frame welded to the cover dips is shown in Fig. 6. When this cover is lowered down on the box, the seating ledge of the box, for the cover is of nickel and has been and is still used as an auxiliary sealing joint and augmented by the liquid lead seal, gives a very effective and hermetically closed container against furnace atmosphere and air. There is some loss of lead by oxidation, this loss can be reduced materially by a charcoal or salt covering over the external surface of the liquid metal. In stationary containers, the making of a lead seal does not introduce

any difficulties since access to the box is always simple before the bell furnace is placed in position. Even during processing, by removing the bell fairly convenient approach to the box is practical for any work or alteration on box or piping or protection of seal.

Attention should also be directed to repairs on resistor heating elements or electrical units which may be taken care of without disturbing the charge in any way. This feature is also incorporated in the heater located under the bottom of box which elements are removable without removal of the box.

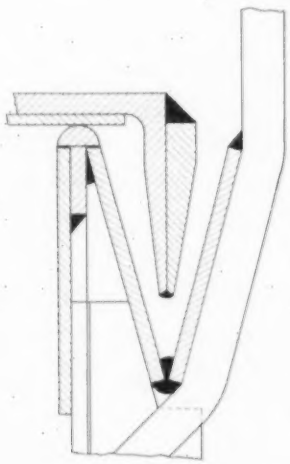


Fig. 6 — Diagram of Lead Sealing Channel for Nitriding Container.

Summary

A summary of the paper definitely establishes the fact that a practically continuous nitriding process has been successfully developed by a judiciously planned layout and the employment of stationary twin unit containers and the use of a removable bell type electric furnace for each unit. Economy of operations and maintenance as well as consistently uniform and selective quality of product are assured by an equipment permitting a practical reproduction of a practice essential to obtain such results.

Conclusion

In concluding, it should be stated that presenting the descriptive nature of the development in nitriding equipment does not permit

a consideration of details of results and physical characteristics obtained from a nitriding practice conducted in such a plant, since it would not serve the purpose of this paper. However, let it be understood that the demand for uniformly high quality of nitrided parts has been met in a decidedly practical manner, economically.

Acknowledgments

Mention should also be made here that this equipment development was largely due to the intense interest in nitriding by Mr. Woodson, manager of industrial heating engineering at our Mansfield plant and T. C. Kelly, sales manager of the industrial heating division at Mansfield, O., and it is to their credit that this paper has been possible at this time.

DISCUSSION

Written Discussion: By J. C. Woodson, manager industrial heating engineering department, Westinghouse Electric and Manufacturing Co., Mansfield, Ohio.

There has been a great deal of discussion upon the subject of nitriding processes and nitriding furnaces in the past two years and there have been a large number of different opinions expressed publicly on these subjects, but these seem to differ. I am confident that we are beginning to approach the time when this process and in general the main components of the equipment will be standardized.

There has not been very much written or said regarding the proper design of nitriding boxes and our experience leads us to believe that this is just as important as either the process or the furnace itself. The latest development in our knowledge along this line, is the development of welded nickel boxes supported in a structural steel frame for rigidity, strength and handling means, and supplied with a flanged cover, which rests in a deep lead seal around the top of the box. This lead seal has in it immersion type electric heaters so that while the box is yet cold, the seal can be brought to a molten state by means of these immersion heaters and the cover seated into the molten lead before the box is placed in the furnace. Likewise, on cooling the box and its contents, the lead can be maintained in a molten state and the cover easily removed without endangering the cover or the box.

I would like to also point out that in the type of furnace described by Mr. Merten, the temperature is controlled both in the heating chamber of the furnace and in the work box itself, thus indicating at all times the temperature both inside the box and outside, which should give the operator very close control of this process.

With the bell type of furnace, as shown, the box and piping connections are permanently installed on the furnace base. These pipes are installed in such a manner that we have not found it necessary to use a circulating fan in-

side the box, as the ammonia inlet pipes are carefully distributed in the box and arranged near the bottom of the charge. The outlet pipes are likewise distributed near the top of the box to carry off the decomposed gases, and from tests so far, this arrangement seems to give more uniform nitriding than the ordinary arrangement using a fan.

Written Discussion: By Robert Sergeson, metallurgical laboratories, Central Alloy Division, Republic Steel Corporation, Massillon, Ohio.

The writer wishes to congratulate Mr. Merten for the valuable information he has brought forward. The five prime factors mentioned for successful nitriding are in accordance with the writer's views and experience.

Stationary nitriding boxes and the continuity of the process are certainly steps in the right direction for production work.

Low melting alloys, e. g. Rose metal and tin-lead eutectic have an embrittling effect on nickel. Whether lead has a like effect, the writer does not know and wishes to ask Mr. Merten if he has experienced or experimented this possibility.

The ammonia piping system appears to present a uniform distribution. However, it has been the writer's experience that positive circulation is essential to satisfactory and uniform nitriding.

H. W. McQuaid:[1] I believe there is something in nitriding which is worth presenting at this time but which has not yet been brought out. I believe the paper by Messrs. Sergeson and Deal is a very clear and complete paper. Most of the papers that have been presented have discussed the ways and means of nitriding in the container and the troubles you are having now. While we have touched upon the metals for the container, I do not know whether all of us realize some of the great difficulties which those who nitride commercially are having. It is a fact that those plants which are doing general nitriding on a large scale have a just complaint. A year or so ago when nitriding just started, there was very little mention made of the difficulties of nitriding containers. Many plants started off in good shape but they are now as we say, up to their necks in trouble because of container difficulties. The fact that you can denitride a container by heating up to 1500 degrees Fahr. is a big advantage in those furnaces in which you can do it. But one particular type is not designed to go over 1200 degrees Fahr., and the only thing to be done in that case is to replace the whole lining, which is expensive. Those which can be heated to 1400 degrees Fahr. and above can be denitrided by heating.

I think Mr. Sergeson's contribution or suggestion of the enamel container is a good one. We are pretty well familiar with the fact that dissociation of the ammonia depends to a great extent upon the surface and in a silicon tube, for instance, dissociation takes place rather slowly when there are no metals present. There is one thing in Mr. Sergeson's paper that struck me peculiarly. He said that the oil seal on the Homo furnace should be carefully made so that none of the oil distills over into the nitriding zone because if it did the result would be that the work would be discolored and perhaps soft. We find it quite the contrary. In fact it was more

[1]Metallurgist, Timken-Detroit Axle Co., Detroit.

or less the results which we had with oil and oil seals and so on which lead to some of the work of the Surface Combustion Company of which considerably more will be said later. In my opinion, a most important contribution for the developing of the nitriding art is found in the report which the Surface Combustion representative, Mr. R. J. Cowan, is going to make. I am particularly interested in this because I have been following it rather closely, and find that his findings can be used with value in all nitriding. We find on batch nitriding that the results are often very erratic. I find that the most important thing in nitriding now (this will explain a lot of the variation which was discussed) is the rate of heating. For successful nitriding it is necessary that the initial nitriding should take place in an atmosphere of practically undissociated ammonia. In other words you should bring the furnace up to temperature so that nitriding takes place before dissociation has appreciably started. If you do that you will find that a lot of the variations and the spotty results with which you are familiar are gone. In that respect I might mention the factors that Mr. Merten has discussed. We have a retort which is very difficult to make ammonia tight and as a rule it leaks so badly that the next department complains of the ammonia fumes and yet we seem to get pretty good results or rather we did at first and the results became gradually poorer as our retort got older. I do not think it was the retort, which was cast Nichrome, as much as the cover which was sheet nickel. Mr. Cowan has developed the fact that it is possible to nitride in a continuous furnace and in doing so he has proved that the condition of the ammonia at the time nitriding starts is the most important factor. Even if you nitride at 1150 to 1200 degrees Fahr. or if you have your dissociation around 80 to 90 per cent, if your initial nitriding ranges, which takes place at about 750 to 850 degrees Fahr. is slow and long enough to give your initial nitrided surface a chance to form in an atmosphere of practically pure ammonia, it is not necessary to worry about removing the hydrogen. As far as I have been able to find out in our furnace you man get the most uniform work I have ever seen and the best results by running it up to 750 degrees Fahr. and holding it for three hours and having your dissociation down around zero, then running it up to 850 degrees Fahr. for three hours, the dissociation increasing to 5 per cent. That was demonstrated last week in a retort which has been in use since April 1928. These results were really the most remarkable I have ever obtained with Vickers hardness as high as 1206 and a surface which is really remarkably tough. Not only that but we seemed to have eliminated any variation of hardness from one end of the box to the other. Our box is a rather crude affair compared to Mr. Merten's and our furnace happens to be a General Electric and not a Westinghouse. However, it works well just the same. We have a rather crude box and no elaborate system of circulating the ammonia. Due to the fact that the distribution pipe was broken, we only had one inlet and one outlet, 8 inches apart in the bottom of the retort. This worked very well provided we brought them up to heat slowly.

I had rather felt that we were losing interest in nitriding and thought

that all the developments might be in furnace refinements and retort refinements as Mr. Sergeson has worked on, but I am quite enthusiastic now and I predict a much brighter picture for nitriding with the development of a continuous furnace which Mr. Cowan will describe to you and the batch furnaces in which we watch carefully the initial nitriding period. This, in my opinion, is most important of all because having once established your nitrided skin, there is very little to worry about.

In regard to the question of packing materials, I want to say that one time we checked thoroughly with Mr. Kinzel his work on nitriding in such compounds as calcium oxide and magnesium oxide and at one time we thought it was the answer. A couple of years ago we were all set on it, but we gave it up because of the difficulty in packing commercially work in powders and so on. But I believe that is going to be an important contribution along with the work which the Surface Combustion Company has done and which Mr. Cowan will now show you.

R. J. COWAN:[2] Mr. Merten's paper is entitled "Nitriding Furnaces and Their Equipment." In this paper he proceeds to describe one furnace that may be used for this purpose. I take it that it is up to me to describe another type of furnace. It was last year at the convention during the Nitriding Session when a desire was expressed for the development of a continuous furnace for nitriding. It was felt that such a furnace might have interesting possibilities and that the next step was to develop such a

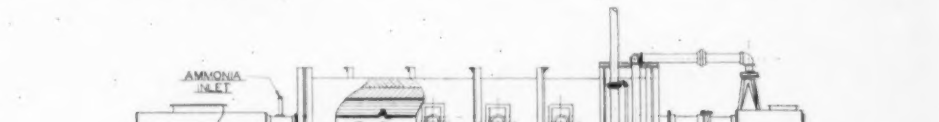
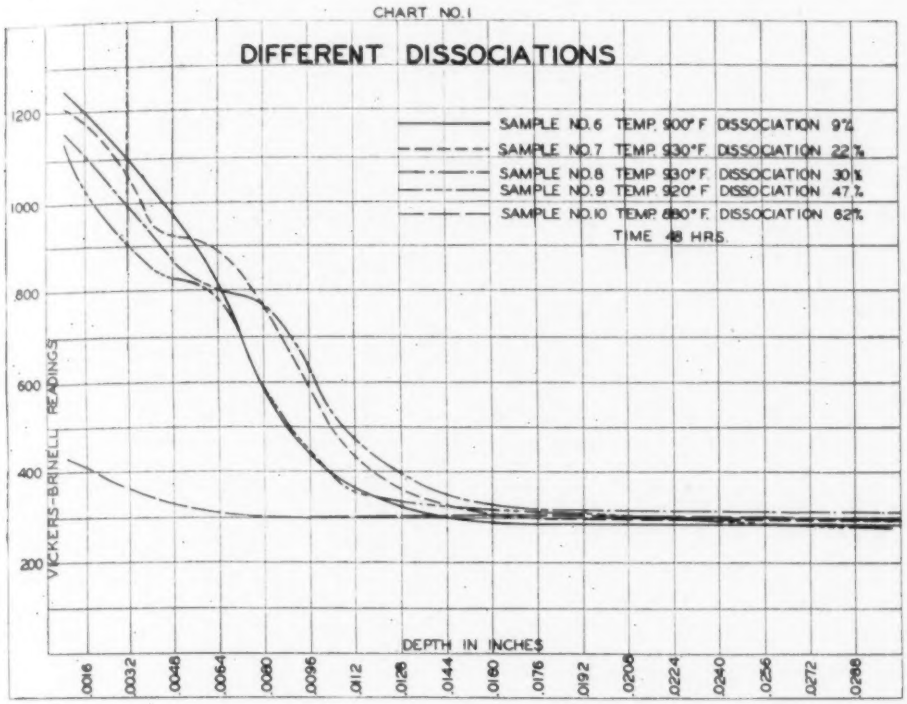


Fig. A.

furnace. The furnace industry accepted this as a challenge. During the year the laboratories of the Surface Combustion Company have been busy working out the details of such a furnace and we are glad to be able to present this information to you at this time. In order to work out the details of a furnace, we found it necessary to make some rather elaborate investigations of the process itself which would have a bearing on the development of a continuous furnace. In our study, we found out some very interesting things about the process and these are what I wish to present now in a preliminary way. The complete paper referred to was published in the October issue of *Metal Progress* so you can get the detailed information there, but I simply want to show you now some of the results we have obtained.

We began our study of this process by trying to find out what would

[2]Metallurgist, Surface Combustion Co., Toledo, Ohio.



happen with different ammonia dissociations. We began from scratch and the first thing we wanted to know was what would be the effect in hardness if we had different dissociations at the outlet of the furnace. You will notice in Chart No. 1 that the dissociations measured at the outlets of the tubes varied from 9 to 82 per cent. It is seen that high dissociation gives very poor results. This shows why in the batch type furnace, it is necessary to maintain the dissociation below 50 per cent in order to get satisfactory results.

If we are going to consider a continuous process, there is one thing of fundamental importance that must be determined; namely, in which direction shall we make the ammonia gas flow through the muffle furnace? If we cause the gas to flow along with the work, then apparently the ammonia dissociation is from low to high. If we cause it to flow counter to the work, then the dissociation is from high to low. In the next chart, chart No. 2, we undertook to study this matter and we chose two temperatures, one at 950 degrees Fahr. and one at 1150 degrees Fahr. One of these curves shows the result from low to high dissociation and the other shows from high to low. In each case the higher curve shows greater hardness and greater penetration and the result of low to high dissociation, both at 950 degrees Fahr. and at 1150 degrees Fahr. The dissociation in these cases was varied from 20 to 82 per cent progressively and then from 81 to 17 per cent over a period of practically 48 hours. This led us to the conclusion that it was desirable in this work to have the ammonia flow parallel with the work.

CHART NO. 2

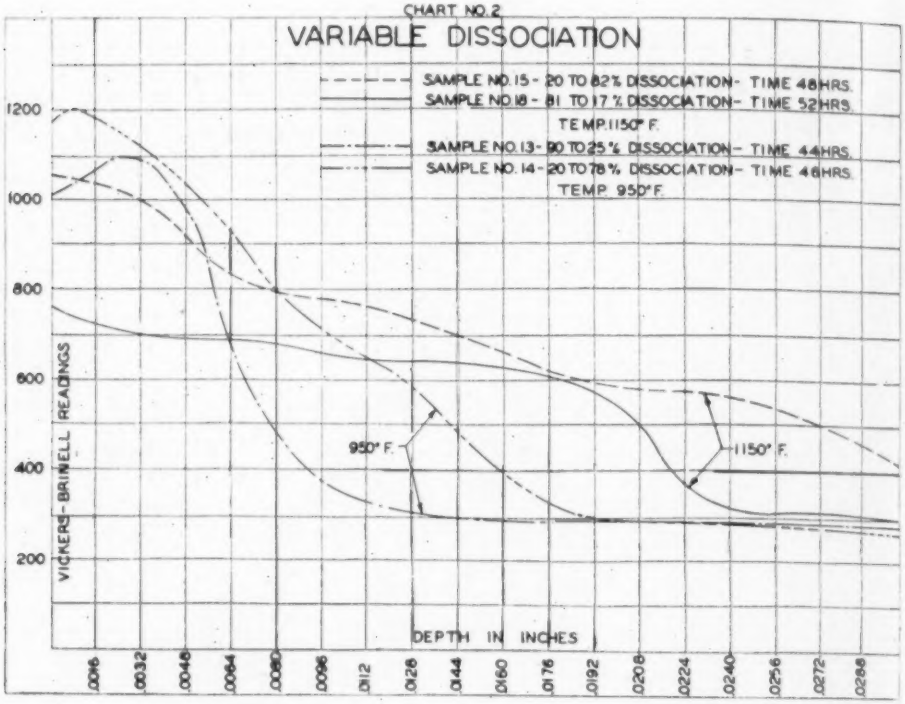


CHART NO. 3

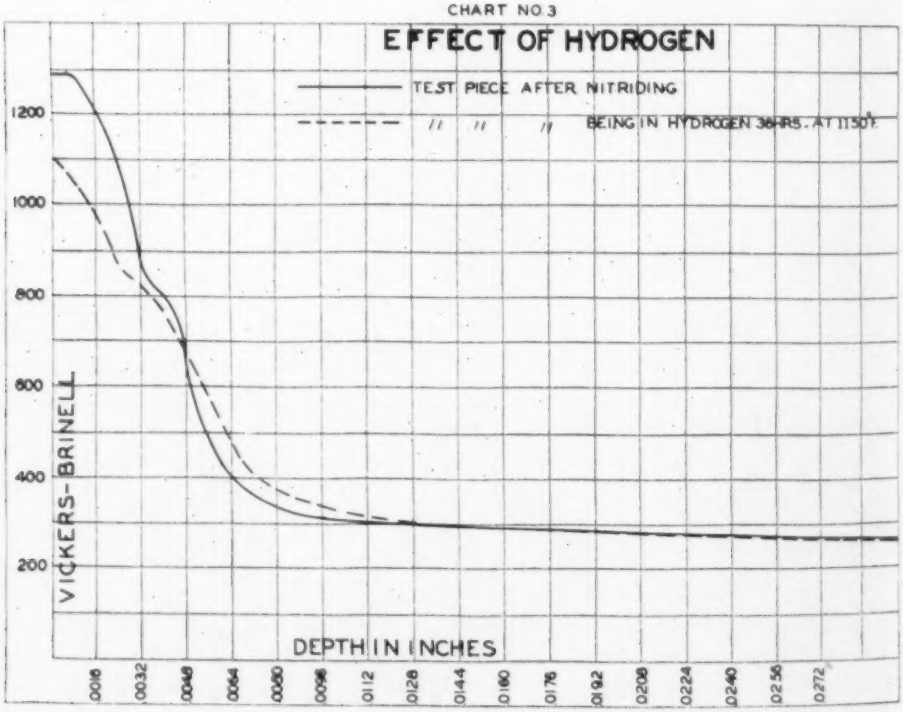


CHART NO. 4

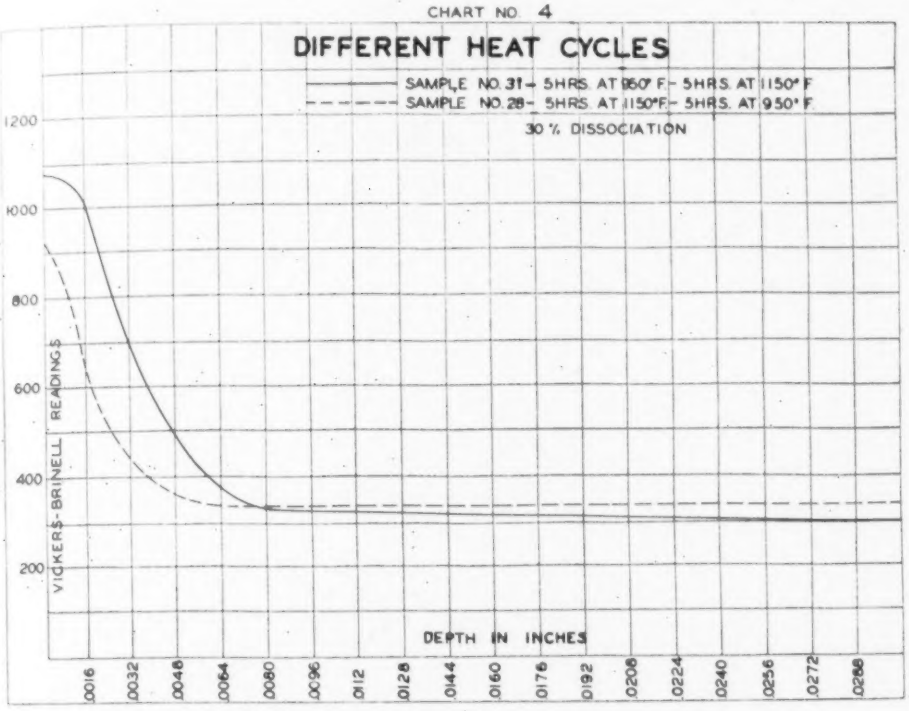


CHART NO. 5

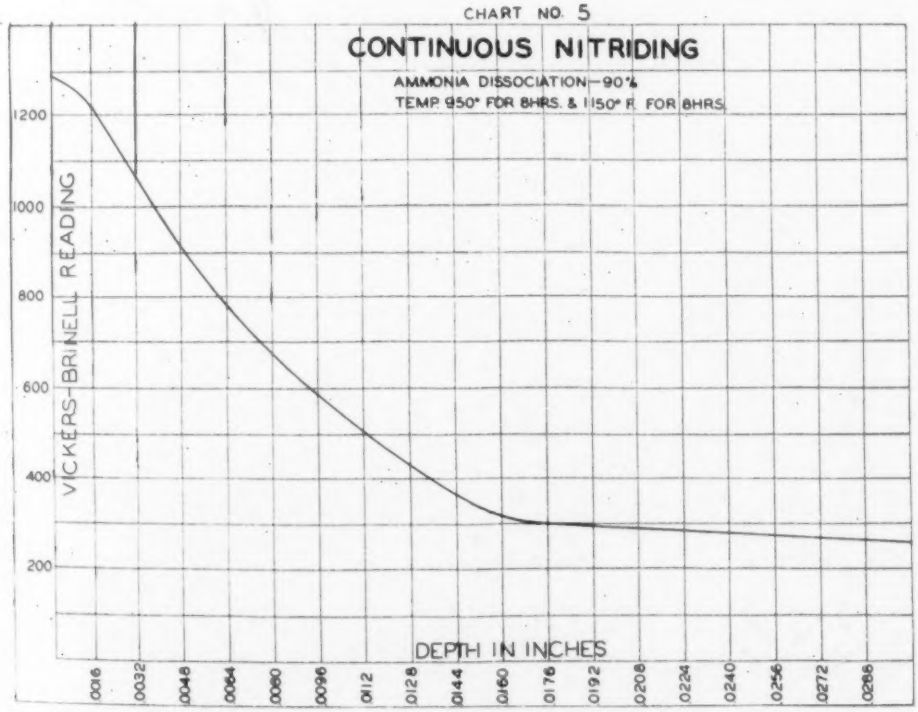


CHART NO. 6

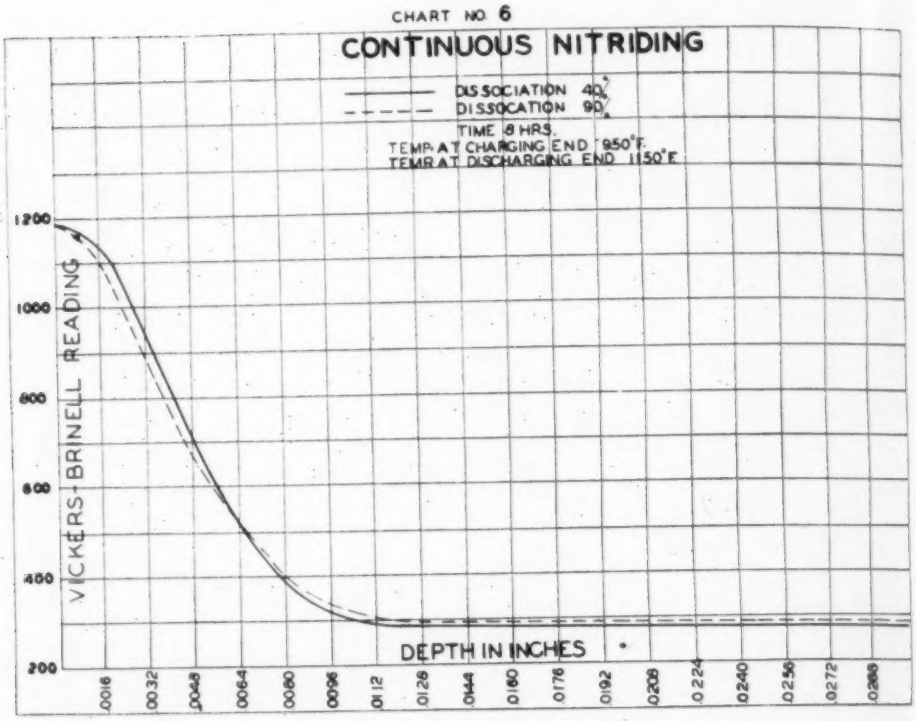
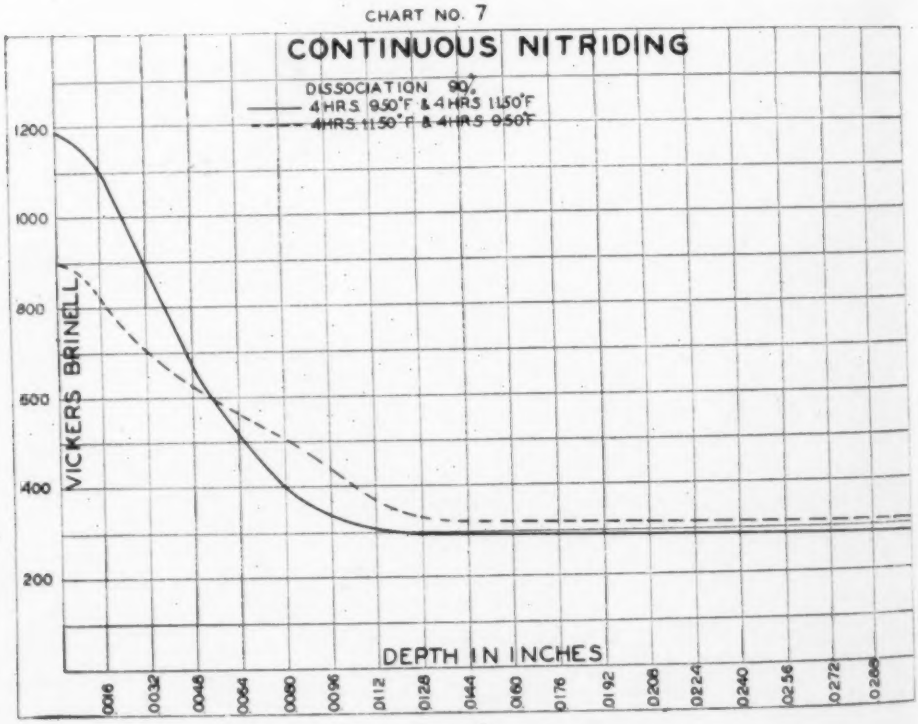


CHART NO. 7



The question then arises as to what effect hydrogen will have on a nitrided case. It is apparent that if we are going to have high dissociation at the outlet of the furnace, necessarily the metal which has been nitrided will be subject to the subsequent action of hydrogen. Chart No. 3 represents one piece which had been nitrided and then subjected to the action of hydrogen at a temperature of 1150 degrees Fahr. for 36 hours. I am told by those who know that the results would have been the same if we had heated the piece in any inert gas for the same time. You will notice a slight decrease in hardness and a slight increase in penetration.

We then attempted to investigate some different heat cycles to see what would happen if we maintained the dissociation constant at 30 per cent. Here is one, chart No. 4, that was held for 5 hours at 950 degrees Fahr. and 5 hours at 1100 degrees Fahr. The solid line shows the results. We then ran a test for the same length of time but reversed the heat cycle in that we held 5 hours at 1150 degrees Fahr. and then 5 hours at 950 degrees Fahr. This work was done to get some idea of what we could expect from ordinary batch type operation.

Chart No. 5 represents our first results with continuous nitriding. You will see in this that the hardness is pretty well up. The ammonia dissociation at the outlet was maintained at 90 per cent. This treatment extended over a period of 16 hours; 8 hours at 950 degrees Fahr. and 8 hours at 1150 degrees Fahr. The hardness curve shown is an average curve of a number of results obtained at the same time. The question naturally arises in considering this as to what our results would have been if, instead of a dissociation of 90 per cent at the outlet, we had maintained a dissociation of 40 per cent. This is shown in detail in chart No. 6. Here the continuous nitriding has extended over a period of only 8 hours. The solid line represents the 90 per cent dissociation and the dotted line represents the 40 per cent dissociation. It is seen that the results are about the same. In chart No. 7 we varied the heat cycle and the dotted line represents the result we obtained by giving the high temperature treatment first.

One very important factor in this whole work is the control that you have over the type of case desired. This is represented by a study of chart No. 7. It would seem that both the hardness and penetration might be controlled by a proper selection of heat cycles. I believe the next year will bring out some very interesting work that has been made possible by study along these lines, and I believe this will furnish one of the greatest possibilities in continuous nitriding in the next year.

In Fig. A, page 516, there is presented an outline sketch of a furnace that we have designed to do this work. A muffle extends through the furnace heated from above and below by gas burners. A liquid seal at the end makes it possible to introduce the work in suitable metal trays which are moved through the furnace progressively by a mechanical pusher. Ammonia inlets at the charging end of the furnace both above and below the work allow the ammonia to move along with the work toward the discharge end of the furnace where the work is removed through another seal and the completely dissociated gas discharged into the atmosphere. It is be-

lieved that this development offers a distinct advantage to the progress of the nitriding art.

Author's Closure

To Mr. Sergeson's lead seal problem I want to give this answer. We have actually had a lead seal in a nickel channel for about two years and that nickel is still good. This probably answers the question of life. We have piping in the box which is also of pure nickel and to date no deterioration has occurred. It is hardly likely that we have obtained an especially pure grade of nickel to explain our success with lead seals on nickel channels. There is a very easy way of protecting the nickel surface from being attacked by lead by coating it with hydro magnesium silicate (talc or soapstone).

Mr. Woodson's timely and well directed remarks on box equipment are appreciated and the emphasis laid upon the importance of correct design of containers is certainly well worth our attention.

In replying to the discussions, I want to again emphasize the fact that our work in the equipment described is beyond the stage where we "seem to get pretty good results," as expressed by Mr. McQuaid, and then have gradually poorer quality to report. Our results are consistently of the characteristics which we specify and no compromise as to quality is ever necessary, and physical dimensions are checked for and are held consistently within specified limits. As to Mr. Cowan's discussion, I am wondering whether an impression is being created that something new and novel in furnaces has been designed. The Rockwell liquid end-sealed conveyor type muffle furnace has been used in bright annealing of copper and copper basic alloys for quite a number of years. A substitution of ammonia (NH_3) for other inert gases and consequent nitriding or cementation with (Fe_2N) is about the only novel feature and change in this method from the age worn copper and bronze conveyor type Rockwell Muffle Annealing Furnace. The adoption of this old design for nitriding can well be appreciated for miscellaneous batch work; however, where due to intricate design such as large crank shafts, a careful alignment of supports has to be provided all through the process—the box conveyor type of support is certainly questionable.

The reference to the experiment of heating nitrided parts in hydrogen for 36 hours as a criterion of what would happen in an atmosphere charged with nascent H, resulting from decomposition of NH_3, is not well chosen and the comparison is not justified.

The experiments to determine the correct flow of gas and heating cycle from a low to a high temperature, are of the same order and as I take it were made in a small stationary laboratory tube furnace to draw conclusions from it to govern the operating cycle of the large muffle furnace is at least courageous. I certainly would like to see the results of a longer operating period of this furnace to become enthusiastic to the same degree as Mr. McQuaid, whose remarks about it indicate that he is sold on this equipment.

NITRIDING THE LARGER FORGINGS

By J. H. Higgins

Abstract

The object of this brief paper is to acquaint the users and prospective users of nitriding steels, in larger forgings, of the care which must be exercised in the preparation of these forgings before nitriding.

The failures of nitriding steels, in many cases, can be attributed to improper preparation of the material before nitriding.

The demand for large nitrided forgings is increasing daily, and there is no reason to believe but that these forgings will find just as much favor, in their respective field, as the smaller forgings.

The brief description of the furnace and box, in which these larger forgings are treated, is interesting, inasmuch as they both depart from what was generally considered correct equipment for the treatment of this steel.

THE art of nitriding steel has been described and discussed in numerous papers presented before the American Society for Steel Treating by authorities far more competent to do so than the writer, consequently, we will only state, briefly, that the steel is a special alloy material, which is forged, heat treated, finish-machined completely, placed in a sealed container, heated to 950 to 1000 degrees Fahr. and exposed, while within this container, to the action of ammonia gas, for varying lengths of time. This procedure puts a hard case upon all surfaces of the steel, ranging from 950 to 1200 Vickers Brinell.

Furnace and Box

The furnace used in hardening the materials described in this paper is one of the largest in service, and departs radically from similar furnaces in that it is oil-fired with six burners; three on each side, with automatic control and a fuel consumption, when at temperature, of one gallon oil per hour per burner. (Fig. 1.)

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, J. H. Higgins, a member of the society, is materials engineer with the Camden Forge Co., Camden, N. J. Manuscript received May 29, 1930.

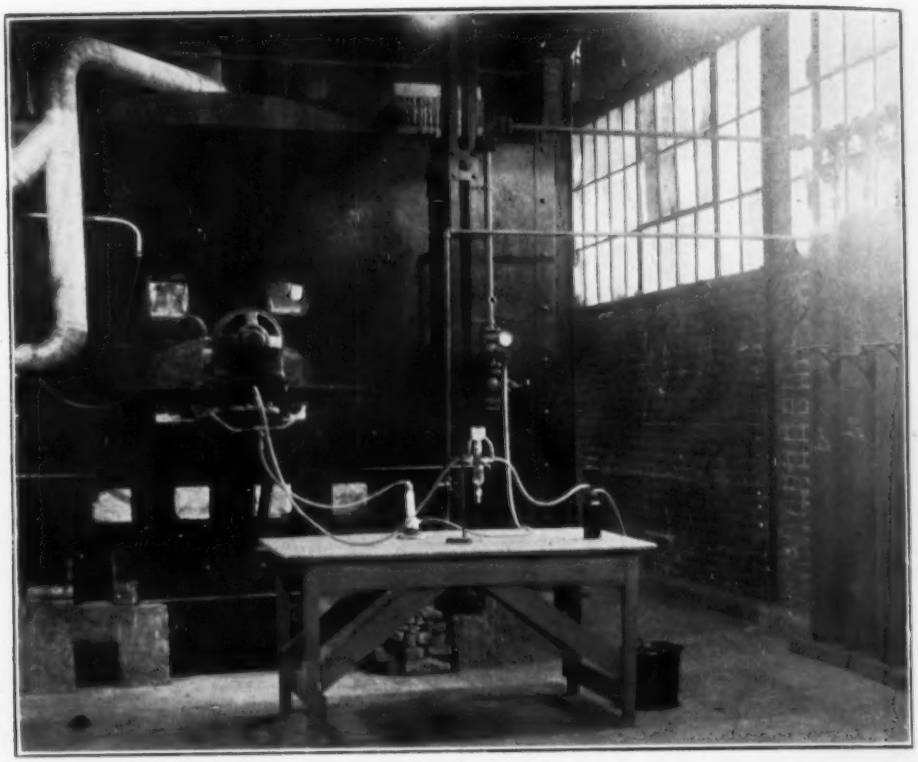


Fig. 1—Nitriding Furnace, Circulating Fan and Dissociation Equipment.

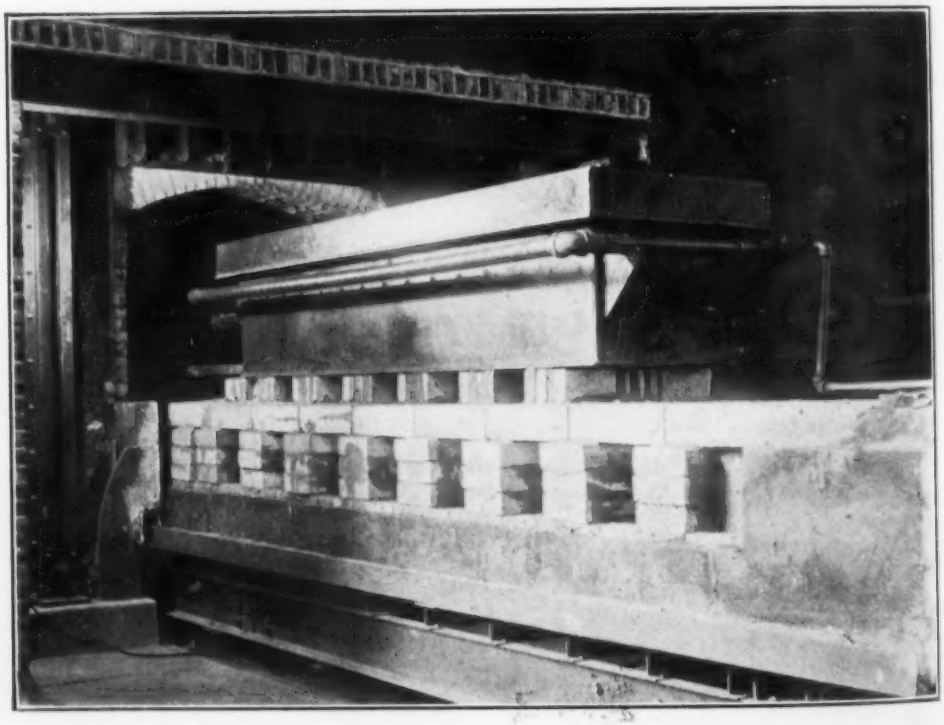


Fig. 2—Charging End of Furnace Showing Nitriding Container on Car Bottom.

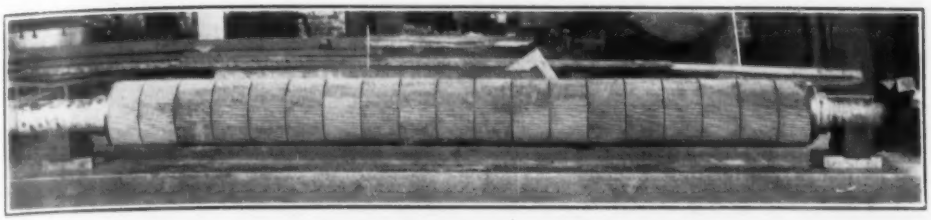


Fig. 3—Corrugated Paper Roll.

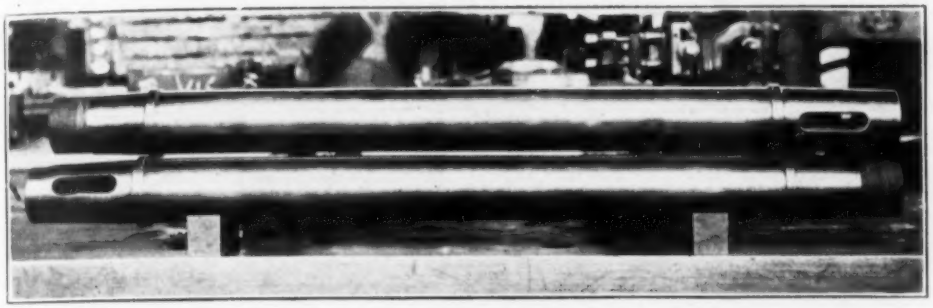


Fig. 4—Locomotive Piston Rods.

The box (Fig. 2) was fabricated from low carbon structural steel, size 36 inches deep; 30 inches wide and 11 feet 7 inches long. This, also, is a departure from some recommended practice, but one which has proven its value; first in cost of material and construction, also in ability to weld all joints air-tight. Test specimens for microscopic examination are taken from the box proper after every heat. This assures an absolute check on the growth and depth of nitrides, and enables one to discard the box before any harm is caused in the nitriding of forgings. This box is equipped with seven recording thermocouples, thus permitting the furnace operator to hold all parts of the box at a constant temperature. The variation, when at holding temperature, can be held within 35 degrees Fahr.

Progress of Nitrided Forgings

The nitriding of steels has increased tremendously in the past year or eighteen months, principally on smaller forgings, and in the past year the designing engineer has reached out and become bold enough to try forgings of a weight and design never before attempted, with the result that it is not unusual today to nitride forgings weighing from 100 to 3000 pounds, and it is these forgings that this paper will briefly discuss.

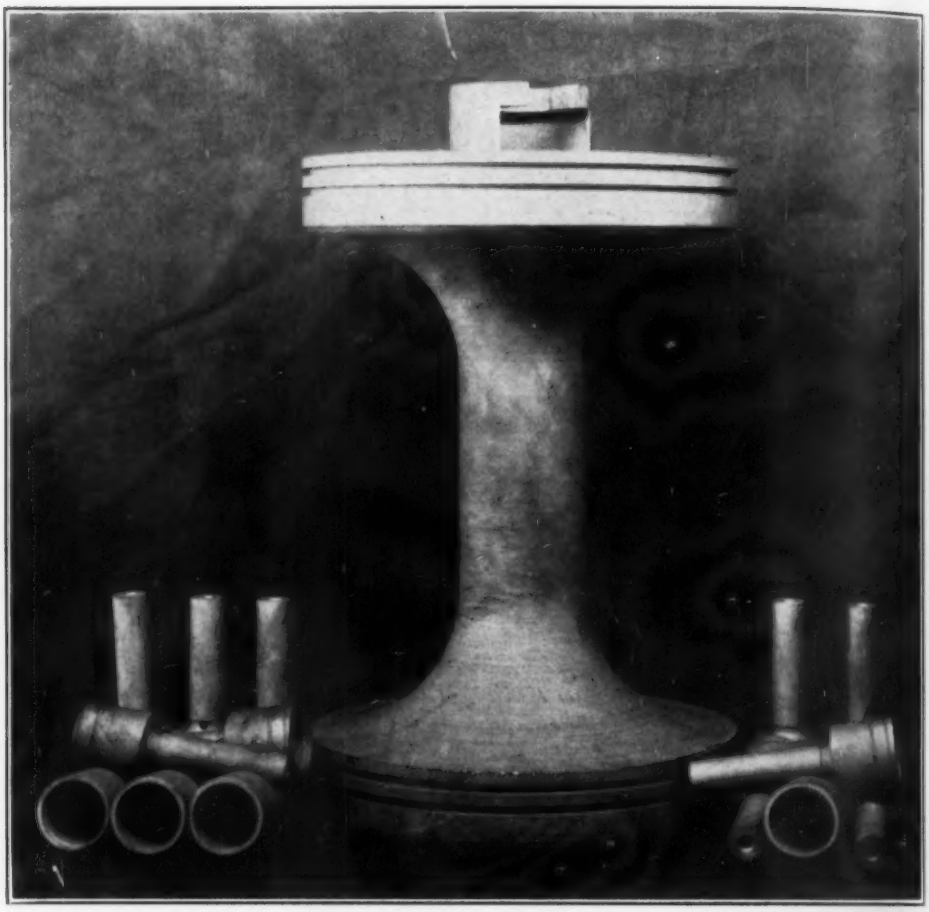


Fig. 5—Large Nitrided Valve.

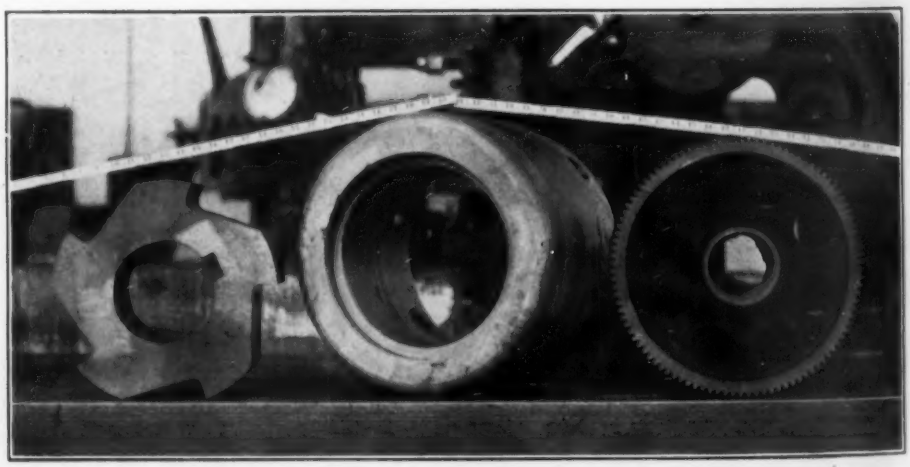


Fig. 6—Nitrided Hub-Ring and Gear.

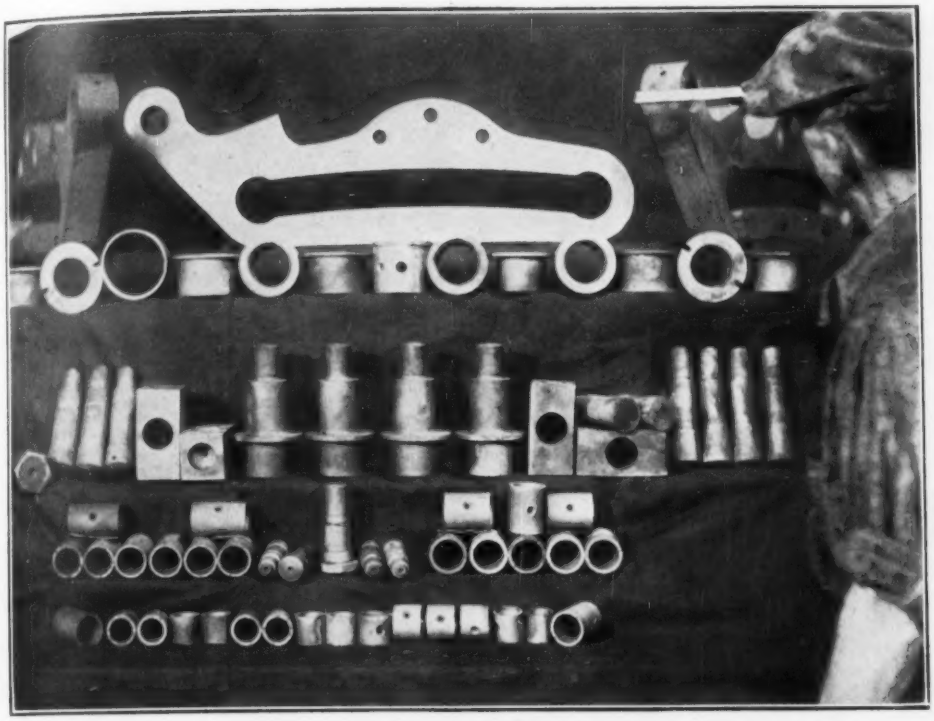


Fig. 7—Locomotive Motion Parts.

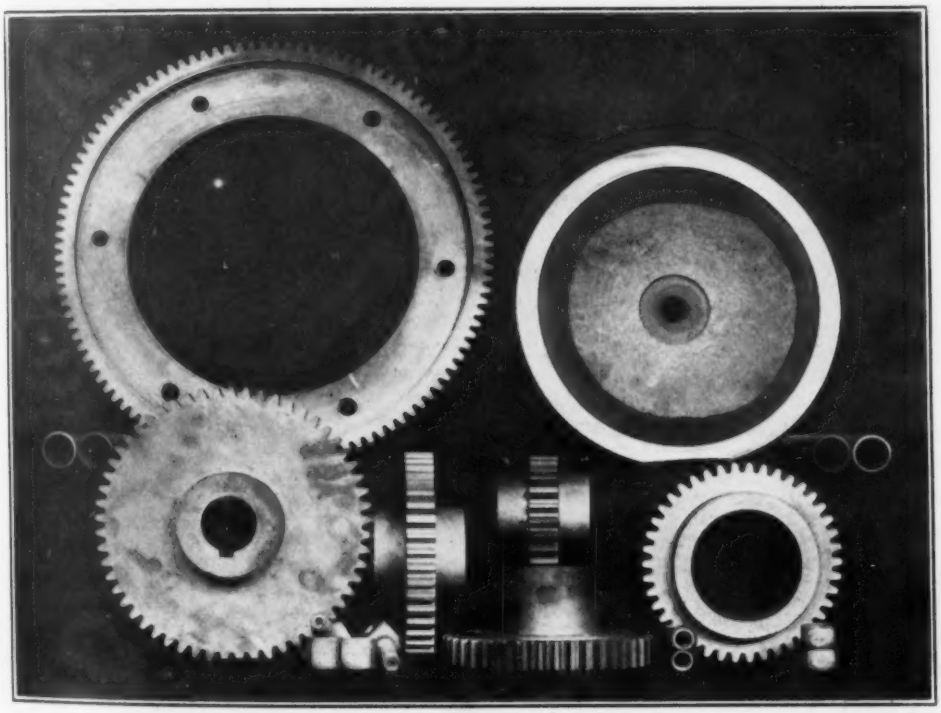


Fig. 8—Large Size Nitrided Gears.

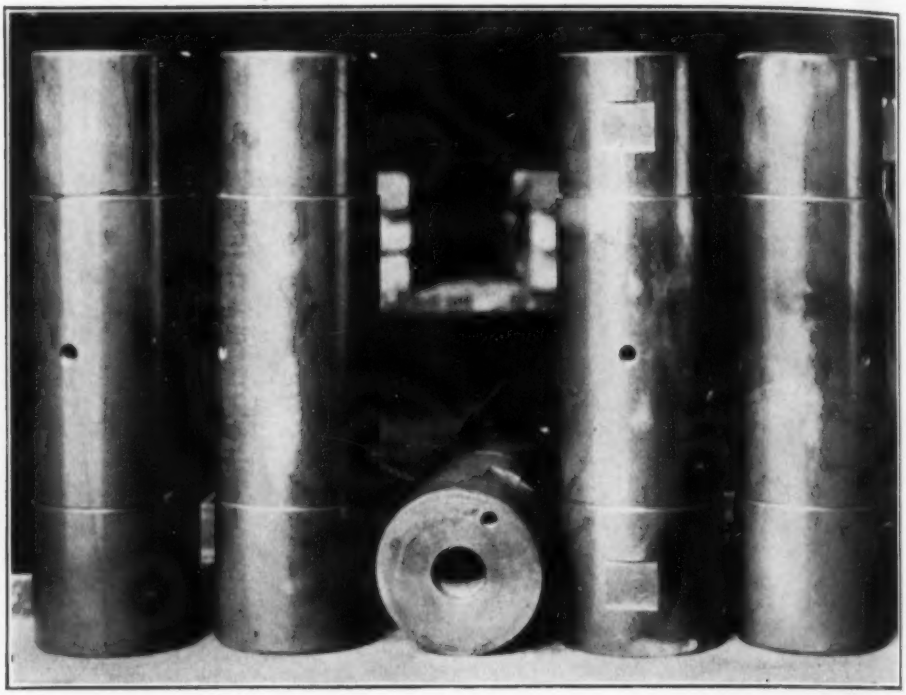


Fig. 9—Large Pins (5½ inches diameter) Nitrided.

From the practical standpoint, there is no difference in the method of nitriding large forgings than there is the nitriding of the small forgings. The same procedure is followed, and almost invariably the same results obtained.

Preparation of Materials

It is obvious, however, that more care must be exercised in the selection of steel and preparation of the larger forgings. Large blooms are more difficult to obtain free of detrimental inclusions which, when present, will interfere with the strength of the core and the uniform hardness of the case. Internal strains in forgings are ever present and more so in the larger sizes. These strains, in many cases, require as many as three normalizing treatments before the final finish machining can be accomplished without distortion.

Extreme care must be exercised in piling large forgings in the nitriding box; forgings should be supported on v-blocks, equally spaced, to prevent sagging. This is of the utmost importance. Even though a distorted forging can be straightened, the question is ever present—will it remain straight and give the satisfaction expected of it in service?

Of the many large forgings in service, there are several which will bear special mention, due to their design and service requirements. Paper mill machines require rolls three to a set. These rolls vary in size from 8 to 12 inches in diameter and 108 to 144 inches long. The service is very severe and the rolls frequently break down after a few months operation; some of these rolls are corrugated, while others are plain. It has been found, through careful calibration, before and after nitriding, that the growth on this type of forging will range from 0.001 to 0.0025 inch with little or no distortion.

Other important forgings are engraving rolls, weighing from 600 to 1000 pounds, Diesel engine piston rods, steam locomotive piston rods, pump shafts, piston heads and many others, some of which are illustrated in this paper. Most of these forgings have been in service more than a year and are entirely satisfactory.

Summary

The nitriding of large forgings can be accomplished with every degree of safety and satisfaction as experienced on small forgings. Care must be used in selection of material, forging, heat treating, machining, normalizing and piling in the box.

Growth appears to be progressive in forgings ranging in size from 3 to 12 inches in diameter and is approximately a quarter of a thousandth for the smaller size to two thousandths for the large size.

No distortion is present if materials are carefully prepared.

The close temperature range in the box assures uniform hardness throughout.

The possible uses of the nitrided steels are limited only by the boldness of the designer and the vividness of his imagination. Whether or not its application to a particular service is practical can be determined only after a thorough engineering discussion, at least. Even this discussion may not clear all minds of doubt; in which event, an actual trial or trials in service are well worth making, for, if the steel is a success, it is a big success and any timidness or hesitancy or any condemnation of the process on account of its newness is not warranted, in view of the possible or probable gains.

It does not follow that every machinery part that wears out should be replaced by a nitrided steel, for there are limitations to its use—but just where the limit is, has yet to be determined.

DISCUSSION

Written Discussion: By Robert Sergeson, metallurgical laboratories, Central Alloy Division, Republic Steel Corp., Massillon, Ohio.

Mr. Higgins' paper is indeed an opportune one for it brings to the industry information of unquestionable value. It is of importance to know that the oil-fired furnace can be used successfully for nitriding if properly designed. Heretofore only gas-fired and electric furnaces were used.

To Mr. Higgins' list of large nitrided parts that have operated successfully, the writer should like to add the following items. Large piston rods 5½ inches diameter x 11 ft. long and 8 ft. long, large locomotive cross-head guides and heavy machine parts weighing as high as 1600 lbs. These have operated highly successfully for 6 months to a year and all are still in operation.

Writer wishes to take exception to the statement regarding the growth occurring in nitriding. From tests which have been repeated several times, we find solid sections have the same growth regardless of cross-section and this growth is dependent upon the depth of the nitrided case (or length of time in nitriding), e. g. a 1-inch round and 5-inch round will each grow 0.002 inch in diameter after being nitrided to a depth of 0.025 inch.

For thin-walled sections, such as bushings, where wall thickness is less than ¼ inch, the growth is best obtained by experience.

Written Discussion: By J. W. Harsch, assistant chief engineer, and J. Muller, development engineer, Leeds and Northrup Co., Philadelphia.

The success obtained by Mr. Higgins is gratifying to those who are vitally interested in the future of nitriding. The author in his short but "meaty" paper has contributed greatly to the practical advancement of nitriding and has also pioneered the field of nitriding heavy work.

The factors which determine the quality of work obtained in nitriding are acknowledged to be:—

(a) Condition of steel before nitriding.
(b) Temperature and its uniformity throughout the load.
(c) Furnace atmosphere.

The state of the material prior to nitriding has not always been considered by the makers and users of nitralloy, to be an influencing factor on the ultimate product. A major part of the failures recorded in the past were due to structure of the material, to decarburized surface, and distortion after nitriding. As the material nitrided is a finished part, very little allowance is made for finishing or lapping. It is essential, therefore, that the material be free from any strains before being placed in the furnace. The nitriding temperature is sufficiently high to bring about a strain relief which has often been erroneously attributed to the operation of nitriding.

The temperatures generally used in nitriding are between 925 and 1000 degrees Fahr. and the possible plastic deformation of the material at these elevated temperatures must be considered when loading the work. Should the placing of the supports in loading be such that the unsupported weight sets up stresses above the creep stress of the material, the work is likely to become deformed. This is especially true when the temperature cycle is 72 hours or more.

The future of nitralloy or any other nitriding steel depends largely upon the wise application of the properties developed in the nitriding operation. Machine parts which are subjected to abrasive wear and slight impact with ample lubrication may be made of nitrided material. Parts subjected to high unit bearing pressures require a deeper case and a high strength core to provide sufficient backing for the case. Some of the early failures of nitrided parts can be attributed to the mis-application of nitralloy. Repeated strain relieving heat treatment such as Mr. Higgins is following to develop the maximum core strength was not generally accepted as necessary by the trade a year ago. The high case hardness will not be of any value if the proper preliminary heat treatments are omitted.

It is comforting to know that more and more thought is now being given to the preparation of the material for nitriding. The preparation work is undoubtedly costly, but the cost is well justified when nitriding expensive machined parts.

Mr. Higgins has shown us that he is well aware that temperature uniformity throughout the work is essential for uniform hardness. A temperature gradient within the load will be responsible for a hardness gradient in the cases obtained as well as a variation in depth of case.

The work temperature, the rate of flow and the distribution of the ammonia within the load determine the characteristics of the case. Natural circulation of the ammonia in the load chamber is not sufficient to give a uniform depth of case. It is therefore necessary to resort to forced and induced circulation.

The use of an iron container in which the nitriding is carried on necessitates very careful check of each load. As soon as the injurious effect of such a container is noticed, it must be renewed. The nitrides formed in the container soon act as a catalyst and deplete the furnace atmosphere of the available ammonia from which the nascent nitrogen essential to nitride the work is obtained.

The work Mr. Higgins has been doing is a distinct step forward in the nitriding field and we are in complete agreement with his statement that "The possible uses of nitrided steels are limited only by the boldness of the designer and the vividness of his imagination."

Written Discussion: By J. P. Walsted, instructor in physical metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

Mr. Sergeson and Mr. Higgins seem to disagree as to the growth of an object when nitrided. The disagreement I believe is more apparent than real. Growth in the diameter of a solid cylinder when nitrided is undoubtedly dependent on case depth and will be the same whatever the original diameter

of the cylinder. Growth of the length of the side will be quite another matter. The growth in this case depends on the depth of case and also on the length of the side. This fact is clearly shown by the corner effect already reported. Growth in hollow cylinders, following the same laws, will cause the bore of the cylinder to become larger if the case depth is sufficient to overcome the natural resistance of the core to expansion. If the case depth is slight, then the resistance of the core material will not be overbalanced and the normal growth of the case will tend to close up the bore of the cylinder. Naturally, there must be a critical ratio between the core thickness and case depth, where growth will not take place in either direction. Where two hollow cylinders from the same nitriding heat show opposite results when measured, a logical procedure would be to examine both to check up on uniformity of case depth.

It is pleasing to see that design of equipment is keeping pace with progress in the process itself. The new nitriding furnaces being placed on the market by furnace manufacturers show that the industry recognizes the importance of the nitriding process. Mr. Merten's remark that no better material than nickel will ever be found for inlet and outlet tubes is somewhat surprising. Such a broad statement is never justified no matter how well developed our knowledge of a product or process. I would like to inform Mr. Merten that monel metal tubes can be used. Also monel metal is not subject to poisoning with long continued use.

A. B. KINZEL:[1] I should like to ask Mr. Higgins if he has made any observation on the relative degree of carburization of the standard nitralloy steels which he is using and the standard chrome vanadium forging steel which I am sure must frequently go through his shop.

Author's Closure

In answer to Mr. Sergeson's question I have always found that there was at least in my experience a direct and progressive growth on the solid forgings. This is the result of many thousand measurements made on forgings, from 3 inches up to 12 inches. I have particular reference to those corrugated paper rolls. There we have nitrided in the neighborhood of 25 of 30 of those and in every case we found practically the same degree of growth. I do not know whether anybody has found anything that is progressive in a ring of any size. There are times that we found out that the rings closed up rather than opened but why I do not know. Take the bushings for instance for locomotive parts, put in 400 or 500 of them in one charge and you will find any number of them have been decreased in the bore size and any number of the bores have increased. Why that is I do not know. In reference to Mr. Kinzel's question—"No." We have never made any comparison on anything other than nitrided steels. I have made some very expensive investigations on the depth of decarburization and in more than one case I have found where the decarburized areas have penetrated better than an eighth of an inch on the circles. That is why I am quite firm in my statement that an eighth of an inch at the very least should be removed from every surface of a forging which is 5 inches or better in diameter.

[1]Associated with Union Carbide and Carbon Research Laboratories, Long Island City, N. Y.

RECENT DEVELOPMENTS IN GAS CARBURIZATION

By O. J. Wilbor and J. A. Comstock

Abstract

This paper discusses the use of city gas in the carburization, bright annealing and heat treatment of steel. The results of the investigations conducted are reported and the effects of the elements composing city gas upon steel pieces are each recorded and their cumulative effect likewise reported.

By controlling the various factors affecting the equilibria of gas mixtures at various temperatures it was found that any hydrocarbon gas regardless of its available carbon content can be used as a carburizing medium by means of the intermittent surge method. Also a hypoeutectoid, eutectoid or hypereutectoid case can be obtained as desired by controlling the active carbon content of the carburizing gas.

THE modern metallurgical operations of heat treating, carburizing, bright annealing, etc., demand a thorough knowledge of the effects of gases on metals and methods of controlling the same. In each of these processes, the work being treated is in a gas atmosphere which must be controlled to give the result desired.

In practice, all the above metallurgical processes use the same basic gas, namely city or manufactured gas. In order to determine the most efficient method of controlling this type of gas, it was necessary to begin with a study of the simple gases which go to make up the gas mixture obtainable today as city gas.

Behavior of Simple Gases

The results of our investigations have brought out the following important characteristics of the various simple gases in the presence of iron or steel at elevated temperatures.

Hydrogen is a powerful reducing gas. It attacks sulphides forming hydrogen sulphide. Steel parts high in sulphur in the

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presence of hydrogen will contaminate other steel parts that are low in sulphur in the same furnace. Hydrogen also attacks carbides forming methane and is a common decarburizer of steel. Steel that is high in carbon in the presence of hydrogen gas will decarburize and form methane which at the same time is capable of carburizing another piece of steel that is low in carbon in the same furnace at elevated temperatures.

Oxygen scales steel below the critical point of ferrite and both scales and decarburizes above the critical temperature.

Nitrogen is neutral in the pure state.

Carbon monoxide reduces oxides and also reacts with iron to form iron carbide.

Carbon dioxide scales and decarburizes steel at heat treating temperatures.

Steam scales steel below the critical point of ferrite, and both scales and decarburizes above the critical temperature, thus behaving much like oxygen.

Methane breaks down at elevated temperatures to hydrogen and carbon and therefore can act on steel to either carburize or decarburize, according to the conditions of equilibrium.

Other hydrocarbons break down in the same manner but are more or less powerful as compared to methane, according to their chemical composition.

Behavior of Mixed Gases

It does not follow that the simple gases will act the same in the mixed state in the presence of other gases as they do alone. The action of a mixed gas depends upon its equilibrium and equilibrium is a function of the following conditions:

1. Volumetric ratio of the component gases
2. Temperature
3. Pressure
4. Rate of flow
5. Character of the components
6. Metal present when they are heated.

The mixed gas with which we are concerned in this paper is what is commonly called city gas with the following average composition:

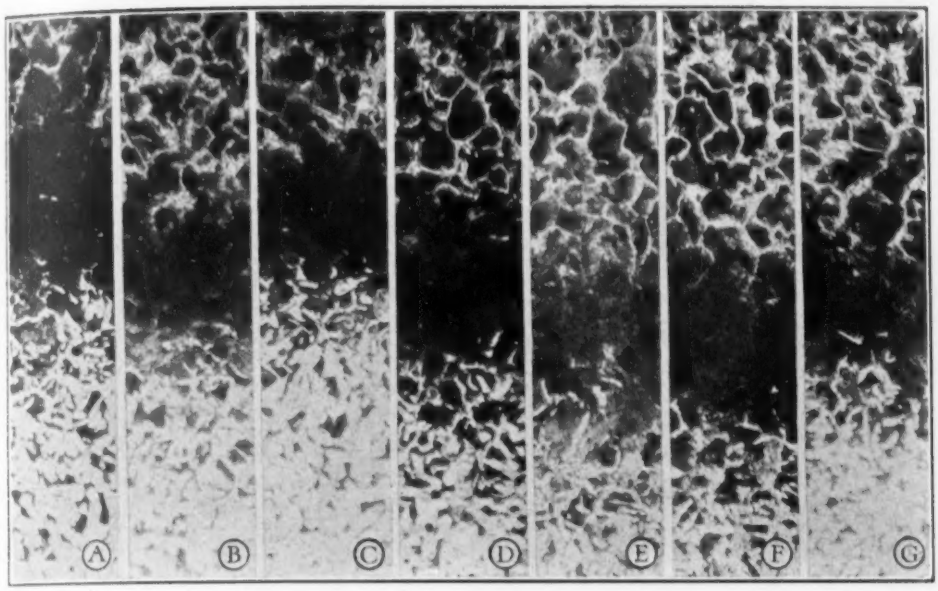


Fig. 1—Armco Iron Carburized with Increasing Amounts of Gas. × 40. See Tests Nos. 162 to 168.

	Per Cent
CO_2	3.0
Illuminants	3.2
Oxygen	1.6
Carbon Monoxide	6.2
Hydrogen	45.0
Methane	25.0
Nitrogen	16.0

Above 1200 degrees Fahr. the hydrocarbons in this gas begin to break down and, depending upon the control of the factors effecting equilibrium, this gas can be made to carburize, decarburize, graphitize or form soot. The details of the control of this gas are brought out in the following practical operations:

Carburizing

Efficient carburization with gas depends upon regulating the dissociation of the hydrocarbons to approximately the same rate as the steel parts can take up carbon or carburize. Dissociation of the hydrocarbons at too great a rate will cause the formation of soot which will deposit on the surfaces of the work to the partial exclusion of the carburizing gas, thus slowing up the carburizing action. Dissociation of hydrocarbons at too slow a rate will cause nonuniform carburization and a low rate of penetration.

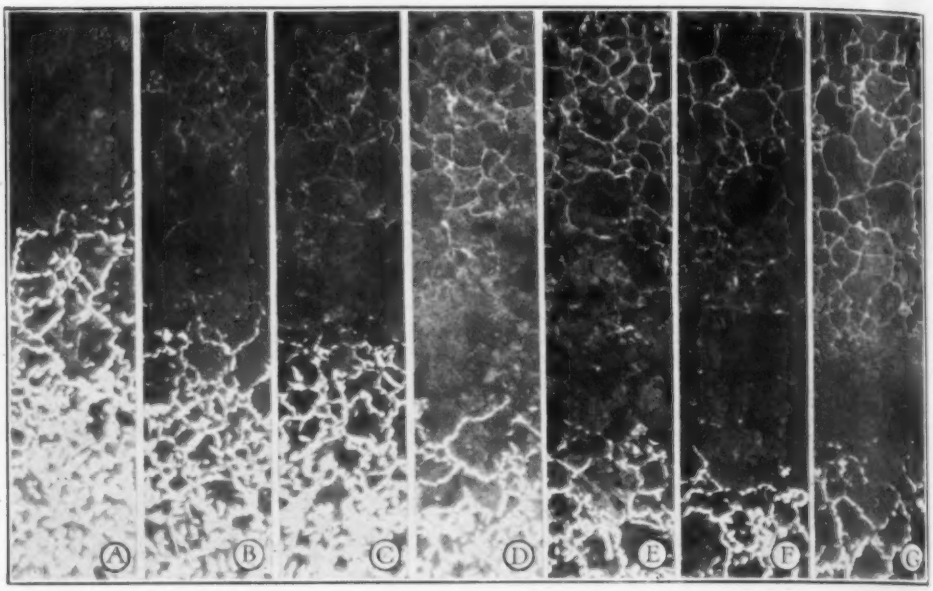


Fig. 2—S.A.E. Steel 1020 Carburized with Increasing Amounts of Gas. × 40. See Tests Nos. 162 to 168.

The equilibrium equation for the dissociation of any hydrocarbon gas is:

$$Cm\ H_{2n\pm2} \rightleftarrows mC + (n\pm1)\ H_2 \qquad (A)$$

From a study of this equation of equilibrium we can state that:

1. Higher temperature speeds up this action from left to right, and there is an equilibrium for every temperature.

2. Pressure slows it down and favors the reaction from right to left.

3. Increase of the rate of flow within certain practical limits increases the amount of carbon formed without changing the equilibrium at a given temperature.

4. The yield of carbon of a unit of gas volume depends upon the composition of the hydrocarbons.

[3 volumes methane (CH_4): 1½ volumes ethylene (C_2H_4): 1 volume propane (C_3H_8): ½ volume benzene (C_6H_6)]

5. Hydrogen slows down the action by favoring the reaction from right to left.

6. Carbon dioxide reacts with the hydrocarbons to reduce the amount of available carbon according to the following equilibrium equation:

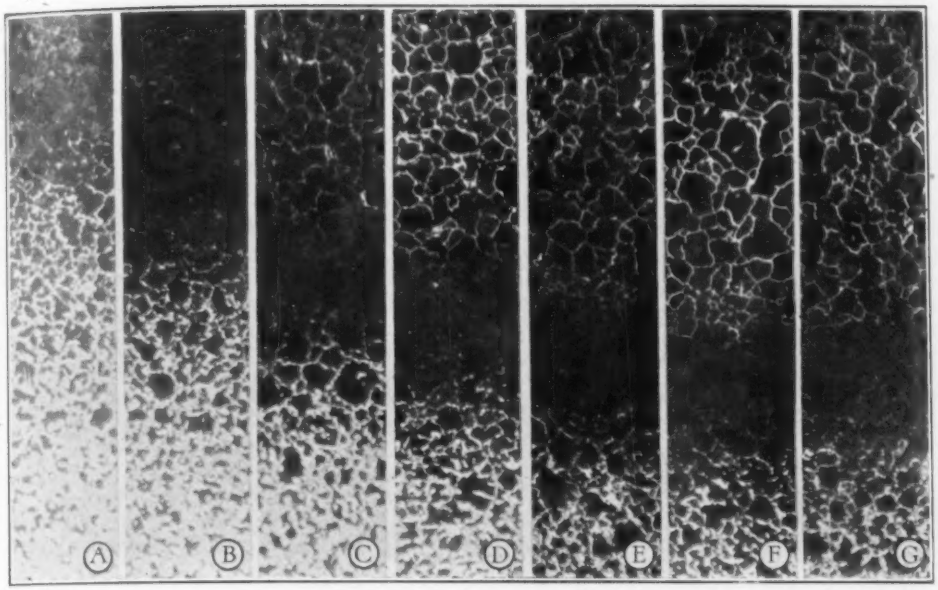


Fig. 3—Screw Stock Carburized with Increasing Amounts of Gas. × 40. See Tests Nos. 162 to 168.

$$Cm\ H_{2n\pm2} + pCO_2 \rightleftarrows (m\text{-}p)\ C + (n\pm1)\ H_2 + 2pCO \qquad (B)$$

7. Carbon monoxide tends to nullify the action of carbon dioxide by favoring the reaction of equation (B) from right to left.

8. Oxygen will produce some products of combustion and reduce the carburizing power.

9. Water vapor acts on the iron in steel according to the following equilibrium equation:

$$xFe + yH_2O \rightleftarrows Fe_xO_y + yH_2 \qquad (C)$$

Small percentages of water vapor retard the dissociation of the hydrocarbons due to the formation of hydrogen at elevated temperatures. Very large percentages of water vapor will form excessive amounts of iron oxide which will greatly interfere with the carburization of the work.

As shown by the equation (A), in the breakdown of hydrocarbons the volume increases so that an increase in pressure would tend to slow down the dissociation by favoring the reaction from right to left. Consequently, more uniform carburization is obtained by using gas at high pressures around 15 pounds per square inch as the dissociation of the hydrocarbons is slowed down allowing

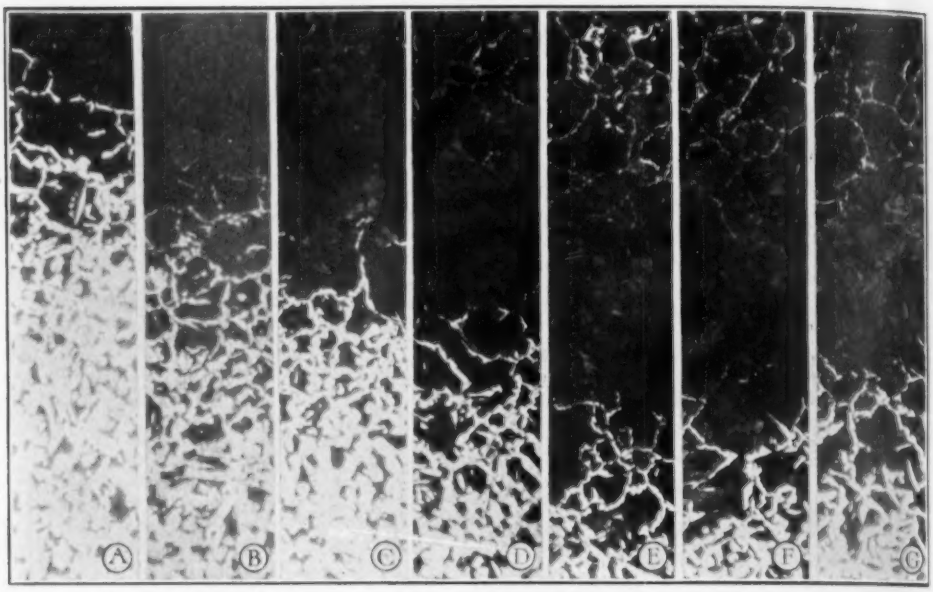


Fig. 4—Guaranteed Normal Carburizing Steel Carburized with Increasing Amounts of Gas. See Tests Nos. 162 to 168.

them to act on the work at a uniform rate from the time they enter the presence of the work until they leave the retort.

Intermittent Surge Carburization

A convenient process for carburizing with hydrocarbons where it is necessary to control the dissociation of these hydrocarbons to give maximum carburizing efficiency with minimum formation of soot is called the intermittent surge method. This process is particularly useful in handling hydrocarbons of a very high carbon concentration, which under the ordinary method of carburizing with gas, would break down to form prohibitive amounts of soot to greatly interfere with the efficiency of the carburizing process. A reduction of the rate of flow of the carburizing gas is inefficient, but a turbulent flow of high speed followed by an interruption for some time overcomes this difficulty. Furthermore, by the use of hydrogen, carbon dioxide or moisture, either alone or together and either admitted continuously to the carburizing chamber or used during part of the time of the interruption of the flow of the carburizing medium it is possible to obtain the desired equilibrium. Hydrocarbons like acetylene, methane, propane, butane, etc., can be used with high efficiency by this process. During the time of interruption

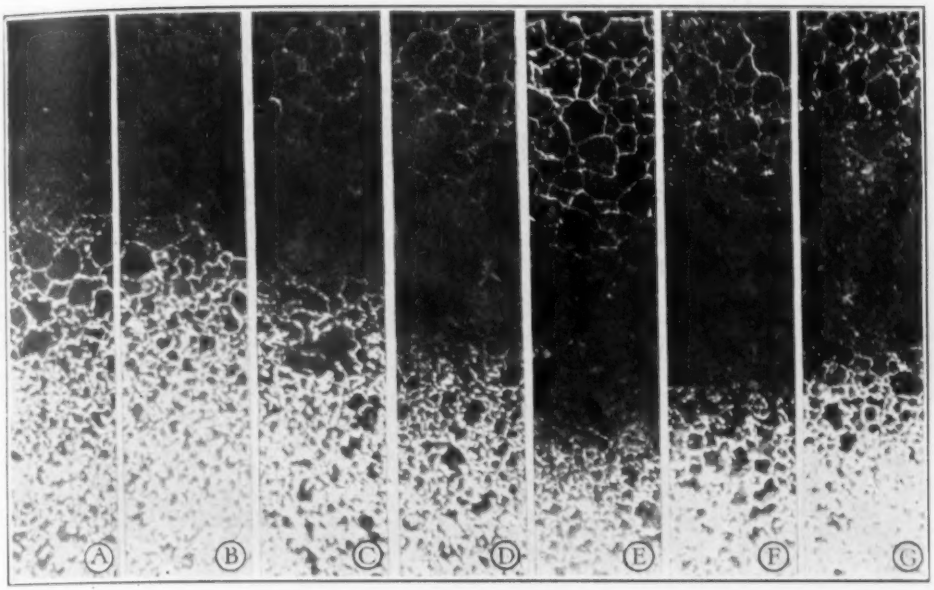


Fig. 5—Jalcase Carburized with Increasing Amounts of Gas. × 40. See Tests Nos. 162 to 168.

of the carburizing gas, the steel will continue to diffuse the carbon from the surface inward and at the same time the surface will be prepared by the secondary gas to be in a receptive condition for the next surge of carburizing.

Table I shows two series of tests, numbers 155 to 168 inclusive, giving data on that part of our research having to do particularly with the use of acetylene and propane gases for carburizing. These tests were carried out in a split-body tube heating furnace connected in a complete gas train making it possible to record measurements of all desired conditions of the carburizing gases before entering and after leaving the presence of the steel specimens held in the tube-furnace at carburizing temperatures. In each of these two series carbon dioxide was used as the dissociation controlling gas. Maximum general efficiency with acetylene gas was obtained in test number 155 using undried acetylene gas intermittently and adding wet carbon dioxide gas continuously throughout the run. The tests numbers 155 and 161 show the variations in results obtainable through control of the total volume of gas used and the length of the time intervals.

Maximum general efficiency with dried propane gas was obtained in test 166 using propane gas for 10 minutes and carbon dioxide gas for 20 minutes alternately throughout the carburizing

Table I

Intermittent Carburization

Carburizing With Gases Rich in Carbon

	Gas Used for Purge					Gas Used for Carburization							
Test Number	Volume (Cu. Ft.)	Kind CO_2	Time of the purge (Minutes)	Pressure	Temperature °F.	Total Volume (Cu. Ft.)	Kind	Measuring Pressure	Measuring Temperature °F.	Surge Pressure Lbs.	Volume per period c.c.	Time of the Carb. Gas Flow Sec.	Length of one period Min.
Carburizing temperature 1800° F.													
155	.075	Wet	10			.1062	Wet C_2 H_2	5.9 inch w. c.	76.33	7½	50	9	5
156	.070	Wet	10			.0850	Wet C_2 H_2	5.9 inch w. c.	74.25	5½	40	7	5
157	.070	Wet	10			.1062	Wet C_2 H_2	5.9 inch w. c.	73.02	7½	50	9	5
158	.075	Dry	11			.1274	Wet C_2 H_2	5.9 inch w. c.	74.02	10	60	10	5
159	.065	Dry	14			.1274	Wet C_2 H_2	5.9 inch w. c.	69.25	5.55	60	12	5
160	.070	Dry	10			.1274	Wet C_2 H_2	5.9 inch w. c.	71.48	10	60	10	5
161	.070	Dry	10			.1593	Wet C_2 H_2	5.9 inch w. c.	74.82	10	60	10	4
162	.07	Wet	15	2.1 inch	78.5	.0176A*	Wet C_3 H_8	22 inch	75.81	6.4	50	7	30
163	.07	Wet	17	2.1 inch	78.5	.0176B	Dry C_3 H_8	22 inch	80.47	6.4	50	7	30
164	.07	Wet	13	2.1 inch	74.0	.02648C	Dry C_3 H_8	22 inch	74.97	6.4	50	7	20
165	.07	Wet	14	2.1 inch	76.0	.0352D	Dry C_3 H_8	22 inch	78.02	6.4	50	7	15
166	.07	Wet	13	2.1 inch	76.5	.0529E	Dry C_3 H_8	22 inch	75.68	6.4	50	7	10
167	.07	Wet	15	2.1 inch	75.0	.0706F	Dry C_3 H_8	22 inch	74.23	6.4	100	14	15
168	.07	Wet	20	2.1 inch	74.5	.1059G	Dry C_3 H_8	22 inch	80.76	6.4	100	14	10

Table I (Continued)

Test No.	Vol. Cu. Ft.	Kind CO_2	Additional Gas Used (Wet CO_2) Measuring Pressure	Temperature °F.	Surge Pressure Lbs.	Vol. per period c.c.	Length of a period Min.	Time of flow of the Add. Gas Sec.	Increase in Weight, Five types of Carburizing Steel, Number 1	2	3	4	5	Total	Analysis of the gas after passing the steel, Per Cent CO_2	Ill.	O_2	CO	H_2	CH_4**	N_2
155	.073	Wet	1.9 in. w. c.	76.33		1 bubble per second			.16046	.14901	.21263	.16126	.17270	.85606	6.4	0.4	0.2	31.2	54.9	1.5	5.4
156	.080	Wet	1.9 in. w. c.	74.25		1 bubble per second			.08662	.08816	.13414	.07160	.07470	.45522	4.9	0.3	0.5	39.3	43.4	2.1	9.5
157	.068	Dry	1.9 in. w. c.	73.02		1 bubble per second			.17814	.15680	.20268	.09041	.09404	.72207	10.0	0.4	1.0	36.8	38.9	2.7	10.2
158	.068	Dry	1.9 in. w. c.	74.02		1 bubble per second			.17347	.10254	.13212	.13464	.13646	.67922	8.3	0.2	0.2	37.6	48.0	3.5	2.3
159	.075	Dry	1.9 in. w. c.	69.25		1 bubble per second			.14491	.16825	.24571	.20339	.17634	.93860	3.2	0.4	0.6	34.8	48.4	3.6	9.0
160	.039	Dry	1.9 in. w. c.	71.48		1 bubble per 2 seconds			.18557	.15621	.16773	.16915	.152271	.83092	13.8	0.2	0.8	31.6	34.9	3.1	15.6
161	.0375	Dry	1.9 in. w. c.	74.82		1 bubble per 2 seconds			.10436	.13577	.18983	.11434	.12183	.66613	7.6	0.2	3.2	25.8	44.3	1.6	17.3
162	.00317		23 in.	78.5	6.4	30	90	7	.14020	.09307	.10287	.08603	.06885	.49102	.70	.57	.71	23.97	57.11	6.22	10.72
163	.00317		23 in.	78.5	6.4	30	90	7	.20269	.11411	.12195	.08693	.08493	.61061	2.44	.56	.80	19.76	58.77	5.02	12.55
164	.00529		23 in.	74.0	6.4	30	60	7	.16140	.15190	.17825	.12305	.11136	.72596	.86	.55	.89	18.58	63.30	4.93	10.89
165	.00634		23 in.	76.0	6.4	30	45	7	.18175	.15963	.16496	.15531	.13720	.79885	1.09	.41	1.10	16.25	58.49	11.87	10.78
166	.0158		23 in.	76.1	6.4	30	20	7	.20074	.18140	.20433	.18469	.16785	.93901	.72	1.08	.60	12.90	58.79	21.64	4.27
167	.02119		23 in.	75.0	6.4	30	15	7	.20021	.18296	.19620	.17446	.15482	.90845	.72	1.24	.42	7.47	68.00	20.55	1.60
168	.0317		23 in.	74.5	6.4	30	10	7	.19132	.17629	.19166	.17635	.14625	.88187	1.03	1.32	.95	11.48	51.02	22.84	11.36

Continuous Addition

*See photomicrographs No. 1-5.

**CH_4 and other saturated hydrocarbons figured as CH_4.

Table II
Quantitative Carburization
Tests Showing How the Concentration of the Case Can Be Regulated

Number of the Sample	Weight in Grams			Kind of Gas	Gas Analysis Before Per Cent							Gas Analysis After Per Cent						
	Before	After	Increase		CO_2	Ill.	O_2	CO	H_2	CH_4	N_2	CO_2	Ill.	O_2	CO	H_2	CH_4	N_2
CG29-4A	38.49134	38.53655	.04521	City Gas / H_2 = 1/2	.8	1.2	1.6	2.6	76.8	8.5	8.5	.4	.4	1.4	4.1	74.6	9.1	10.0
CG29-4B	39.25040	39.29310	.04270															
CG30-4A	39.34695	39.38270	.03575	City Gas / H_2 = 1/2	1.0	1.2	1.6	3.0	74.25	9.68	9.27	.4	.4	1.0	4.4	73.3	10.1	10.4
CG30-4B	38.41080	38.44690	.03610															
CG47-4A	39.62780	39.67420	.04640	City Gas / H_2 = 1/2	.6	1.2	1.6	3.2	75.9	8.7	8.8	.8	.2	1.6	5.0	71.0	9.84	11.56
CG47-4B	39.35570	39.40390	.04820															
CG31-4A	39.20335	39.24520	.04185	City Gas / H_2 = 1/1	1.4	1.6	1.4	3.4	70.2	12.5	12.5	.4	.2	1.2	6.0	69.4	11.8	11.0
CG31-4B	40.86760	40.90960	.04200															
CG32-4A	40.02810	40.08630	.05820	City Gas / H_2 = 1/1	1.2	1.6	1.4	3.9	68.0	13.2	10.7	.8	.4	1.2	5.0	68.0	13.2	11.2
CG32-4B	39.46140	39.51710	.05570															
CG33-4A	38.82890	38.89490	.06600	City Gas / H_2 = 2/1	1.8	2.0	1.4	4.7	62.4	17.34	10.36	1.0	.6	1.4	6.0	61.5	17.75	11.75
CG33-4B	37.17740	37.23070	.05330															
CG34-4A	39.87105	39.90340	.03235	City Gas / H_2 = 2/1	1.8	2.2	1.6	4.2	62.7	16.7	11.2	.8	1.2	1.4	6.2	62.2	15.85	12.35
CG34-4B	39.76295	39.79650	.03355															
CG35-4A	39.57170	39.62615	.05445	City Gas / N_2 = 1/2	.6	1.0	1.4	1.6	15.25	9.9	70.25	.4	...	1.0	2.8	17.2	9.06	70.54
CG35-4B	40.19855	40.25180	.05325															
CG36-4A	40.07247	40.15390	.08143	City Gas / N_2 = 1/2	.8	1.2	1.0	1.8	15.65	9.52	70.03	.8	.3	.5	2.4	16.4	8.67	70.93
CG36-4B	40.79750	40.88560	.08810															

Table II—Continued

				City Gas	95													

Table II—Continued

CG37-4A	38.62635	38.66065	.03430	$\frac{\text{City Gas}}{CO_2} = \frac{95}{5}$	7.2	3.0	1.8	6.4	44.2	25.45	11.95	5.6	.8	1.8	7.4	43.3	26.3	14.9
CG37-4B	40.87355	40.91265	.03910	$\frac{\text{City Gas}}{CO_2} = \frac{95}{5}$	7.0	3.2	1.4	6.0	42.8	25.7	13.9	4.8	.4	1.0	9.0	43.33	25.35	16.12
CG38-4A	38.37010	38.40610	.03600	$\frac{\text{City Gas}}{CO_2} = \frac{94}{6}$	8.2	3.2	1.2	5.8	40.8	25.8	15.0	3.4	...	.8	12.6	44.85	22.40	15.95
CG38-4B	39.68810	39.71280	.03470	$\frac{\text{City Gas}}{CO_2} = \frac{93}{7}$	9.8	3.0	1.6	5.8	41.15	23.85	14.80	4.1	.5	.8	13.4	46.17	19.9	15.2
CG40-4A	39.49995	39.54295	.04300	$\frac{\text{City Gas}}{CO_2} = \frac{94}{6}$	7.2	3.2	1.6	6.2	45.4	22.9	13.50	3.4	.5	1.5	13.0	49.5	19.1	13.0
CG40-4B	38.81680	38.85370	.03690	$\frac{\text{City Gas}}{CO_2} = \frac{93}{7}$	8.6	3.2	1.4	6.2	43.0	23.4	14.2	3.8	.2	1.1	12.8	46.4	20.85	14.85
CG39-4A	39.57207	39.60745	.03538	$\frac{\text{City Gas}}{\text{Flue Gas}} = \frac{2}{1}$	4.0	2.3	1.1	4.2	31.3	17.15	39.95	2.4	.2	.4	8.8	33.8	14.8	39.6
CG39-4B	40.13500	40.16599	.03099	$\frac{\text{City Gas}}{\text{Flue Gas}} = \frac{1}{1}$	5.8	1.8	.8	3.0	21.4	15.8	51.4	4.6	.2	.7	6.9	25.7	12.3	49.6
CG46-4A	38.91910	38.98855	.06955	$\frac{\text{City Gas}}{\text{Flue Gas}} = \frac{1}{1}$	5.8	1.6	1.0	3.2	24.85	13.95	49.60	3.4	.4	1.0	5.6	26.13	13.5	49.97
CG46-4B	40.25275	40.32435	.07160	$\frac{\text{City Gas}}{\text{Flue Gas}} = \frac{1}{1}$	5.8	1.2	1.4	3.2	24.1	14.6	49.7	2.6	.2	1.0	6.0	28.6	13.1	48.5
CG48-4A	39.22820	39.26848	.04028	$\frac{\text{Wet City Gas}}{\text{Flue Gas}} = \frac{3}{2}$	5.0	2.2	1.0	3.8	27.3	16.0	44.7	2.6	.2	.8	8.6	31.73	13.17	42.90
CG48-4B	39.79790	39.83420	.03630															
CG41-4A	38.03845	38.09745	.05900															
CG41-4B	40.01618	40.07265	.05647															
CG42-4A	39.01265	39.03695	.02430															
CG42-4B	38.54428	38.57160	.02732															
CG44-4A	39.78370	39.84680	.06310															
CG44-4B	37.24740	37.32578	.08238															
CG45-4A	39.10250	39.10180	.00070															
CG45-4B	40.73325	40.73315	.00010															
CG43-4A	37.54295	37.58715	.04420															
CG43-4B	40.73910	40.79155	.05245															

Table II—Continued

Number of the Sample	Depth of Case (Millimeters) Hyper eu.	Eutec-toid	Hypo eu.	Total d.	Furnace Temper-ature °F.	Meter Readings Before Cu. Ft.	Meter Readings After Cu. Ft.	Initial Volume Cu. Ft.	Meter Readings Before Cu. Ft.	Meter Readings After Cu. Ft.	Volume After Passing Cu. Ft.	Increase in Volume Cu. Ft.	Initial Pressure Inches	Pressure After Passing Inches	Initial Temperature °F.	Temperature After Passing	Time of Test Hours
CG29-4A	....	.363	.957	1.320	1725	70.140	73.740	3.600	.773	4.421	3.648	.048	1.4	.3	80.0	84.0	3
CG29-4B																	
CG30-4A	....	.330	.660	.990	1725	75.305	77.105	1.800	5.970	7.748	1.808	.008	.9	.2	82.0	85.5	3
CG30-4B																	
CG47-4A	....	.165	.495	.660	1725	42.400	45.400	3.000	70.737	73.754	3.017	.017	1.1	.25	83.0	89.0	5
CG47-4B																	
CG31-4A	....	.330	.660	.990	1725	79.800	81.600	1.800	10.389	12.242	1.852	.053	.8	.2	85.0	90	3
CG31-4B																	
CG32-4A	.330	.330	.660	1.320	1725	80.050	86.650	3.600	13.950	17.610	3.660	.060	1.6	.35	84.0	88.0	3
CG32-4B																	
CG33-4A	.220	.440	.660	1.320	1725	88.400	92.000	3.600	18.016	21.696	3.680	.080	1.3	.35	85.0	90	3*
CG33-4B																	
CG34-4A	....	.221	.924	1.145	1725	94.050	95.860	1.800	23.070	24.921	1.851	.051	1.2	.2	82.0	88.0	3
CG34-4B																	
CG35-4A	.440	.127	.528	1.155	1725	96.700	98.500	1.800	25.750	27.601	1.851	.051	1.3	.2	84.0	91.0	3
CG35-4B																	
CG36-4A	.617	.264	.759	1.640	1725	99.500	103.100	3.600	28.343	32.023	3.680	.080	1.6	.75	86.0	91.0	3
CG36-4B																	
CG37-4A	....	....	.990	.990	1725	4.100	7.700	3.600	33.009	36.671	3.662	.062	1.2	.5	83.0	87.0	3*
CG37-4B																	
CG38-4A	....	....	.330	.330	1725	94.400	96.200	1.800	38.365	40.229	1.864	.064	1.1	.2	90.0	94.0	3*
CG38-4B																	
CG40-4A	....	.264	.551	.815	1725	15.708	17.508	1.800	44.827	46.786	1.959	.159	.9	.25	87.0	90.0	3
CG40-4B																	
CG39-4A	....	.033	.627	.660	1725	12.410	14.210	1.800	41.414	43.381	1.967	.167	1.1	.25	85.0	88.0	3
CG39-4B																	
CG46-4A	.335	.160	.495	.990	1725	38.400	41.400	3.000	66.972	70.341	3.369	.369	1.6	.3	82.5	86.0	5*
CG46-4B																	
CG48-4A	....	....	.660	.660	1725	46.300	49.300	3.000	74.117	77.359	3.242	.242	1.3	.25	83.0	87.0	5
CG48-4B																	
CG41-4A	.410	.415	.330	1.155	1725	18.520	20.320	1.800	47.242	49.124	1.882	.082	1.0	.3	81.0	85.0	3
CG41-4B																	
CG42-4A	....	.165	.165	.330	1725	21.700	23.500	1.800	50.509	52.355	1.846	.046	1.6	.25	82.0	87.0	3
CG42-4B																	
CG44-4A	.660	.330	.660	1.650	1725	28.000	31.600	3.600	56.286	60.007	3.721	.121	2.2	.5	82.0	87.0	3
CG44-4B																	
CG45-4A	....	....	....	.....	1725	33.400	37.000	3.600	61.746	65.552	3.806	.206	2.1	.5	81.0	86.0	3
CG45-4B																	
CG43-4A	.725	.660	.495	1.880	1725	26.000	27.800	1.800	53.290	55.184	1.894	.094	1.7	.3	82.0	88.0	3
CG43-4B																	

*Sample Sooted.

process. Tests numbers 162 and 168 inclusive show the results obtained by varying the rate of flow. (See photographs Figs. 1 to 5).

Quantitative Carburization

In practice, in many cases, it is important to be able to carburize steel parts to arrive at a certain definite carbon concentration for a given case depth. Heavy duty parts, such as large gears, are desired to be free from excess iron carbide in the form of cementitic network, since this is a common cause of pitting and spalling in service. It has been customary to first carburize such parts to obtain the proper depth of case, regardless of the high carbon concentration obtained on the surface, and to follow the carburizing process with a diffusion process which would drive the excess carbon inward and leave eutectoid concentration on the surface. Such a practice, of course, is expensive.

In other cases it is desirable to carburize with the maximum carbon concentration at the surface with the minimum case depth. This is true of thin sections which have to be glass hard on the surface and yet retain a good percentage of their section as a tough core to get away from excessive brittleness of the part as a whole.

By the traditional method of carburizing using a solid carburizing medium, it was impossible to control any of the factors which would give either a maximum case depth with a eutectoid carbon concentration at the surface, or a minimum case depth with a maximum carbon concentration at the surface. About all that could be expected from such a method was to obtain a eutectoid concentration for a case depth of less than 1/32 inch and a hypereutectoid concentration for a case depth in excess of 1/32 inch. However, the modern gas carburizing process makes it possible to control all the factors affecting the case and makes it entirely practical to arrive at a given carbon concentration and case depth without the necessity of diffusion. Furthermore, it opens up a new field for application of carburizing to parts of thin sections requiring maximum surface hardness.

Sykes[1] has shown that there is a given equilibrium between a hydrocarbon and a steel at a given temperature. Therefore, city gas must be able to carburize hypoeutectoid, eutectoid or hyper-

[1]W. P. Sykes, Transactions, American Society for Steel Treating, Vol. 12, 1927, p. 737.

eutectoid cases when a controlling gas is added to the city gas which will influence the hydrocarbon dissociation, as shown in equations (A), (B), and (C). Such gases are carbon monoxide, hydrogen, water vapor, nitrogen, and dry flue gas.

Table II shows four series of tests using hydrogen, nitrogen, carbon dioxide and theoretical dry flue gas as the dissociation control gases respectively in connection with city gas as a carburizing medium.

Maximum case depth for eutectoid concentration using a guaranteed normal carburizing steel was obtained in test CG29-4A and -4B which uses a mixture of one part of city gas to two parts of hydrogen in the dry condition.

Tests numbers CG29-4A to CG34-4B inclusive show the variation in carbon concentration to be obtained by controlling the rate of flow of the carburizing medium as well as by varying the proportion of city gas and hydrogen in the different mixtures.

Tests numbers CG40-4A and -4B, using the dry mixture of 94 parts of city gas and 6 parts of carbon dioxide give nearly as deep a eutectoid case.

Tests numbers CG37-4A to CG48-4B using carbon dioxide as the controlling gas shows the variation in carbon concentration that can be obtained by controlling the rate of flow and the proportion of carbon dioxide and city gas in the carburizing medium.

Tests number CG36-4A and -4B using a dry mixture of one part of city gas to two parts of nitrogen gave the maximum carbon concentration. City gas diluted with nitrogen will produce a shallow case in a short length of time having a high carbon concentration at the surface. Such a diluted gas makes it possible to carburize with small quantities of the carburizing medium and at the same time a high rate of flow, thereby producing uniform carburization throughout the retort. This procedure can also be applied in the case of hydrocarbons of high order of carbon concentration to control their dissociation which under the ordinary method of gas carburizing would form excessive amounts of soot.

Tests CG35-4A to CG36-4B inclusive, show variation in carbon concentration produced by varying the rate of flow of the carburizing medium.

Tests numbers CG43-4A and -4B using a dry mixture of three parts of city gas to two parts of theoretical flue gas shows maximum carbon concentration. This result compares with that obtained where

nitrogen was used as a dilutant and is what would be expected from a mixture high in nitrogen such as flue gas is.

Tests CG41-4A to CG45-4A, inclusive, show the variation in carbon concentration obtained by varying the rate of flow of the carburizing medium. It is interesting to compare tests CG44 and CG45 since the only difference in the two tests was that the latter was not dried. Table II shows that whereas in the dry condition this gas mixture gave very excellent results, however in the wet condition carburizing was practically nil.

Summary

It is found that by controlling the various factors affecting the equilibria of gas mixtures at various temperatures that the following desirable results may be obtained.

a. Any hydrocarbon gas regardless of its available carbon content can be used efficiently as a carburizing medium by means of the intermittent surge method.

b. A hypoeutectoid, eutectoid, or hypereutectoid case can be obtained as desired by controlling the active carbon content of the carburizing gas.

Acknowledgment

The authors wish to take this occasion to express their indebtedness to R. G. Guthrie for his inspiring guidance throughout this work, and to J. Chumasero for his excellent metallographic assistance.

DISCUSSION

Written Discussion: By P. C. Osterman, vice president, American Gas Furnace Co., Elizabeth, N. J.

This paper is very interesting and adds considerably to our knowledge of what takes place in carburizing when gas is employed as the carburizing medium. It is easy to see that it represents a great deal of work and serious effort, and very careful study is required to fully appreciate the results obtained.

No doubt, the carburizing temperature employed in making the tests was determined upon in order to speed up the work as much as possible. We rather regret that it was found necessary to do this inasmuch as in general practice lower temperatures are used and as results at such lower temperatures would not coincide with those obtained in the test. Lower temperatures are generally used, as we all know, as with them a better grain structure can be obtained in the work with less warpage and distortion as a result of quench-

ing. Lower temperatures also result in longer life for the containers, furnace lining, etc.

An extensive series of tests was conducted in a rotary carburizing machine employing propane gas as the carburizing agent. With this gas, undiluted in any way, a very heavy, undesirable carbon deposit formed in the retort; however, a good case was obtained. Subsequently, tests were made using various gases to dilute the propane. This series of tests included mixtures of propane and steam, propane and charcoal producer gas, propane and nitrogen, also a mixture of propane and air which gave equally as good results, if not somewhat better than any of the other diluents with which the propane gas was tested for carburizing. Air being free, it follows that it is recommended for diluting the gas. Naturally, our company has patented this process of carburizing.

Some irregularities which were encountered in making early commercial applications of this process were thought to be due to the fact that a critical temperature exists below which much slower carbon penetration was obtained than by using higher temperatures; however, various tests failed to confirm this opinion. It is, however, just one more reason why we would have preferred to see the tests made by Messrs. Wilbor and Comstock conducted at a lower temperature.

The method of supplying gas to the retort in surges is an interesting one and it may be that the condition so obtained will warrant the complication of providing for furnishing the gas or mixture of gases to the retort in this fashion.

In other series of tests which were conducted by us, small amounts of powdered compound were supplied to the retort by mechanical means at definite intervals. Excellent results were obtained and the carburizing time was apparently reduced below other methods for obtaining a given case depth. It might also be mentioned that only a very small amount of compound is required by this method. We must admit, however, that results were apparently equally as satisfactory when a slow, continuous feed of powdered compound to the retort was provided.

While the tests just mentioned did not duplicate the conditions described in the paper under discussion, they do cause us to wonder whether the intermittent method of supplying gas to the retort would prove sufficiently advantageous to warrant its application commercially.

The control of concentration will, in our opinion, prove one of the greatest advantages of gas carburizing. The carbon content can also be adjusted by adjusting the ratio of air to propane and feeding the mixture continuously to the retort. Where undiluted natural gas is used as the carburizing agent, what might be regarded as a modified form of the surging system in which only a single surge occurs, consists in allowing a period at the end of the run when no more gas is added to the retort. During this period the carbon in the case migrates to give a lower concentration on the surface with a greater depth of penetration grading off gradually to the core.

Another very interesting field for research in the use of various gases or mixtures of gases for carburizing lies, in our opinion, among the catalyzers. It is our firm opinion that heat resisting alloys at least to some extent influence favorably the rate of carburizing. We believe, however, it should be possible

to find some means of speeding up the penetration of the carbon into the steel by simply having present in the retort a limited amount of some salt or metal or by adding a gas to act as a catalyzer. By means of such an agent we consider that there are possibilities of speeding up the rate of carburizing to the same extent that the process has been speeded up over the old method of using a plain charcoal by the addition of one of the various so-called energizers.

We would like to repeat that we consider the paper just presented by Messrs. Wilbor and Comstock a most excellent one and we feel that it contains a great deal of material worthy of further very careful study by all of us interested in this process.

It may not be out of order for us to state at this time that, in our opinion, even though carburization is rather old, there are possibilities for great forward strides in this art and that gas carburizing holds great promise for the future. What is ordinarily termed pack carburizing is also really gas carburizing, for gas does the carburizing whether the gas is generated in the container or in a separate producer.

We believe that the time will come when virtually all carburizing will be done by means of gas, that is, gas introduced as such to the carburizing retort.

A. B. KINZEL:[1] This is a particularly interesting paper and certainly should be acknowledged as a very fine piece of work. The relations which Messrs. Wilbor and Comstock have pointed out are particularly interesting. There was one point mentioned in Mr. Osterman's discussion which I feel the authors have not sufficiently emphasized. They have shown that with a given CH_{n+2} definite results can be obtained with a definite practice. The point that they have not emphasized and which I wish to emphasize is that this given CH_n must be constant. If it varies then it is necessary to vary the other conditions to match it and that, of course, would be difficult indeed. Mr. Osterman mentioned propane. That is a single, definite, specific gas that does not vary. It may be that the authors did not think it necessary to emphasize this point because their findings may show that illuminating gas as it is usually found is sufficiently uniform. However, we do know that it must be uniform within certain limits in order to work the scheme which they advocate.

D. LEWIS:[2] Mr. Wilbor and Mr. Comstock are to be congratulated on presenting this paper and I hope they will start a new interest in gas carburizing similar to the attention at present directed to nitriding. There are several manufacturers in the country who are gas carburizing, and a number of problems require solving. I would like to suggest to the authors that what I believe we want from gas carburizing is a means of putting into the surface of the material to be carburized as much carbon as it will dissolve at the temperature of the carburizing, in other words, if we can saturate the surface skin with carbon then our rate of carburizing is fixed by the rate of diffusion. You cannot carburize any faster than that. Then the control of the carbon content in the surface in the finished material can be easily obtained by giving a period

[1]Associated with Union Carbide and Carbon Research Laboratories, Long Island City, N. Y.

[2]Research engineer, American Chain Co., Bridgeport, Conn.

at the end of carburizing during which diffusion only takes place and in that way allowing the surface concentration of carbon to fall down to whatever figure is desired. The same result could also be obtained by intermittent carburizing. The difficulty is to know how to be sure of conditions that will saturate the surface of the material with carbon. Presumably a gas which will deposit carbon at the temperature of carburizing is one which is capable of delivering up enough carbon as far as equilibrium conditions are concerned to fully saturate the steel, because if it will liberate solid carbon it will not have any difficulty in giving out sufficient carbon to saturate the steel. The difficulty seems to be not in the composition of the gas but the means of getting the gas to the surface of the material and in that respect I am not sure that we are not getting into some of the same difficulties that are experienced in nitriding. I very much hope that Mr. Comstock will do more work and perhaps supply the answer to some of these problems. I would like to ask him whether he has any knowledge of practical application of the intermittent method of supplying the gases. That seems to be one of the most interesting features of his paper, and I would be very much interested to know whether he has any actual works practice of that type of carburizing.

Authors' Closure

Dr. Wilbor and I are very grateful to Messrs. Osterman, Kinzel and Lewis for their interesting and helpful discussions. I will try to answer them as briefly as possible. Mr. Osterman brings up the question of the carburizing temperature, which, as the tables show, is 1800 degrees Fahr. Dr. Wilbor and I have investigated the effect of the variation of temperature upon these equilibria conditions and we find that the equilibrium varies with the temperature, but we also find that the maximum carburizing results as far as laboratory practice goes is obtained at 1800 degrees Fahr. We conducted all the other tests at that same temperature for purposes of comparison.

Mr. Osterman mentions the practical process of diffusion as being a special case of the intermittent surge method of carburization. The advantage I can see, of the intermittent method over diffusion method, is that with the diffusion method it implies that your carburizing medium is cut off at the end of the carburizing cycle and the carbon is allowed to diffuse further into the steel during a sort of quiescent period with no flow of gas over the steel. You probably have noticed that the equilibrium of the gas is a touchy proposition and the rate of flow is most important. Consequently, if you shut off the rate of flow of gas, conditions change from carburizing to decarburizing. We do not believe it is controllable under these conditions to arrive at a definite carbon concentration on the surface by cutting off the carburizing gas. However this diffusion process has been in practice for some time. There is no getting around that. We are looking at this thing from the angle of further developments in carburization.

One feature of the intermittent method, of course, is that it actually speeds up the rate of carburization due to the beneficial effect of the secondary gas in preparing the surface of the steel for further carburization. The diffusion method does not do this.

Mr. Kinzel brings up the very important question of variation in the analysis or type of hydrocarbon gas used with special reference to city gas which, as you know and which I mentioned, is a mixture of gases. That is a very important academic consideration and one to which the authors have given considerable thought. I am glad Mr. Kinzel brought it out. However, for practical purposes and , as far as the laboratory tests are concerned, there were other features much more important regarding the eventual results of carburization, namely, moisture content. That one is much more important than several others put together and as a result we tried to arrive at the maximum efficiency of the mixture of gases we used. Mr. Lewis brings up the question of the control of the carburization medium being used—that is the rate of flow being increased up to the point where soot is formed. That is just exactly what we tried to do. In other words we tried to get the maximum carburization from the gas and that is at the point where soot is formed because beyond there soot obstructs carburization.

In regard to the surge method, what I wanted to bring out is that the surge method does another thing beside controlling the equilibrium of the gas atmosphere, and that is a chemical effect it has of cleaning the surface of the parts being carburized of any depleted carburizing gas.

I want to thank you again for your kind consideration of our paper.

THE MODIFYING ACTION OF FERROSILICON UPON THE CARBURIZATION PROCESS

By E. G. Mahin and F. J. Mootz

Abstract

It has been shown by earlier researches in this laboratory that by mixing finely ground ferrosilicon with an ordinary solid carburizer, the velocity of carbon absorption by the iron or steel piece is reduced. This action made possible the use of a higher temperature for carburization, the retarding action of ferrosilicon reducing the slope of the carbon gradient, thus preventing building up a hypereutectoid outer case and the separation of free cementite upon slow cooling. The use of higher temperatures increases the velocity of penetration of the absorbed carbon and results in an important saving in the time required to produce a case of specified depth.

This paper is a report of further work, designed to provide an explanation of the modifying action of ferrosilicon. Experimental work shows that the effect is the result of a surface action, since the efficiency of the ferrosilicon increases with decrease in particle size. It is concluded that ferrosilicon reacts to some extent with carbon monoxide, also that silicon itself is carried to the steel piece and there dissolved in the surface layers, thus probably in two ways reducing the rate of carbon absorption. The carrying action is thought to be due to the formation of volatile sulphides of silicon, this by reaction of ferrosilicon with hydrogen sulphide, which is nearly always present in the gases evolved from commercial carburizers.

IN an earlier report[1] it was shown, as a result of experimental observations, that certain ferro-alloys produce important effects upon the depths and character of case, obtained when the finely divided alloys are mixed with ordinary carburizing compounds before the steel or iron pieces are packed in the carburizing pots. Special at-

[1]Mahin and Spencer, "Depth and Character of Case Induced by Mixtures of Ferro-Alloys with Carburizing Compounds," Transactions, American Society for Steel Treating, Vol. 15, 1929, p. 117.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. Of the authors, who are members of the society, Dr. E. G. Mahin is professor of metallurgy at Notre Dame University, Notre Dame, Ind., and F. J. Mootz is connected with the research laboratories of the Republic Creosoting Co., Indianapolis. Manuscript received June 21, 1930.

tention was given to the effect of ferrosilicon, which was found to be that of reducing the per cent of carbon in the extreme outer case, thus making possible the use of temperatures for case carburization, higher than would otherwise be practicable without the formation of free cementite during cooling in the pot. It was proved also that during such treatment, even when there was no direct contact between steel or iron and ferrosilicon, there was an addition of dissolved silicon to the exterior layers of the piece, although analytical evidence on this point was not as extensive as was desired.

A review of earlier work bearing upon this problem was given in the paper already cited. This may be summarized in the statement that various investigators have shown that the presence of elements other than carbon, already present in the steel itself, affects the rate at which carbon is absorbed during case carburization, as well as the rate of penetration of carbon so dissolved (silicon, in particular, retarding both actions), and that iron or steel may be cemented with such elements as silicon, nickel, chromium and others, by heating the pieces after packing in the ferro-alloys of these elements, or in the finely divided elements themselves. It was further shown by Sanfourche[2] that cementation with silicon could be accomplished by heating the piece in a stream of silicon chloride.

It is, of course, a well established fact that silicon, present in a steel in more than traces, raises the thermal transformation points, lowers the pearlite ratio for carbon, and diminishes the solubility of carbon in iron[3 to 11].

The possibility of such cementation by other elements than carbon has inspired a number of inventors in taking out patents

[2]*Comptes rendus,* Vol. 183, 1926, p. 791.

[3]Charpy and Grenet, *Engineering,* Vol. 73, 1902, p. 626.

[4]Wust and Peterson, *Metallurgie,* Vol. 3, 1907, p. 811.

[5]Hague and Turner, *Journal,* Iron and Steel Institute, Vol. 82, 1910, p. 72.

[6]Grenet, *Comptes rendus,* Vol. 150, 1910, p. 921.

[7]Lake, *Iron Age,* Vol. 84, 1912, p. 81.

[8]Charpy and Cornu-Thenard, *Journal,* Iron and Steel Institute, Vol. 91, 1915, p. 276.

[9]Swartz, Payne and Gorton, *Transactions,* American Institute of Mining and Metallurgical Engineers, Vol. 69, 1923, p. 791.

[10]Swartz, Payne, Gorton and Austin, Ibid., Vol. 68, 1923, p. 916.

[11]Becker, *Journal,* Iron and Steel Institute, Vol. 112, 1925, p. 239.

covering their processes[12]. In other cases the addition of metallic oxides to the carburizer has been made for the purpose of increasing the $CO:CO_2$ ratio within the pot by catalytic action, thus increasing the rate of carburization[13].

It is worthy of note that in none of these processes has it been proposed to accomplish anything in the direction of controlling the character or depth of the carbon case. In all cases except the last one cited, the idea has apparently been to produce a case on the steel piece which would be essentially a high carbon alloy steel, and no note was made of any alteration in the rate of absorption or penetration of carbon.

The results of the investigations of Mahin and Spencer[1] appeared to suggest the possibility of practical application in the field of commercial carburization since, especially by use of ferrosilicon in the carburizer, the time necessary for the production of a case of given depth could be materially shortened by raising the temperature of carburization, without thereby producing hypereutectoid outer case zones and consequent brittleness of the surface layers. The mechanism of the action was not entirely clear but it was suggested that the proved solution of silicon in the outer layers of the piece during carburization reduced the rate of carbon absorption at a given temperature, thus permitting higher temperatures with consequent increase in the rate of carbon penetration.

The manner in which silicon is added when the ferro-alloy is in direct contact with the piece is sufficiently obvious, but similar addition was noted when no such mechanical contact was permitted. The suggestion of the authors that silicon hydride is the carrying agent was not tested, and it will be shown in the present report that this theory is untenable.

In order to obtain some further information which might serve to clarify the mechanism of the action, as well as to check some of the earlier conclusions concerning this action, the work embodied in the present report was undertaken.

Comparison of the Influence of Some Silicon Alloys Elementary Silicon and Ferroboron

Armco ingot iron rods were turned on the lathe to approximately

[12]See Ackerman: German 79,428, Bates: German 83,093; Lecarme: French 327,984; Fennell: British 114,446; Allen and Christian: U. S. 1,226,739.

[13]Hodson, "Report on the Grönwall Process," *Chemical and Metallurgical Engineering*, Vol. 28, 1923, p. 308.

½ inch diameter and finished with a smooth and bright surface. These were cut into 1½ inch lengths and carburized under the conditions noted.

The carburizing material was that used in earlier work—a commercial grade, energized with barium carbonate and a small amount of calcium carbonate. This material was received from the manufacturer in the form of small pellets. It was ground for use and screened, the fraction saved being that passing the 20-mesh sieve and retained on the 200-mesh.

The ferro-alloys and silicon were each ground to pass the 200-mesh sieve. Each mixture of these with the carburizer was thoroughly rolled on paper and packed in the nickel-chromium carburizing pot with as little disturbance as possible, in order to minimize segregation.

Ferroboron—This material was selected on account of the close chemical relation between boron and silicon. No analysis was made to determine the per cent of boron in the compound.

Silicon—The material used was a technical grade, shown by analysis to contain 95.8 per cent of silicon and 1.18 per cent of iron.

Ferrosilicon—This was the 50 per cent grade, as used in many earlier experiments, and shown by analysis to contain 49.4 per cent of silicon.

Magnesium-silicon—Elementary silicon and magnesium, both in the powdered form, were mixed in atomic proportions and heated in a hard glass tube until the reaction was completed. The resulting product was a pale blue powder. According to the equilibrium diagram for the magnesium-silicon system, this product should consist of about two-thirds eutectoid of Mg_2Si and silicon, and one-third of the primary phase, silicon.

After the pots had been packed and the covers luted, they were placed in the electrically heated muffle furnace at room temperature and allowed to come up to temperature with the furnace. These runs were made at 1740 degrees Fahr. (950 degrees Cent.), the time consumed in coming to this temperature being somewhat more than four hours. The furnace was held at the temperature stated above for five hours, when the switch was opened and the pots allowed to cool with the furnace. The temperature was maintained, in this and in all other cases, by means of control pyrometers.

Transverse sections of the treated pieces were prepared for microscopic examination and case depth measurements were made by

Table I

Effect of Alloys and of Silicon upon Character of Case, when Carburized for 5 Hours at 1740 Degrees Fahr. (950 Degrees Cent.)

Constituent Added	Per Cent of Added Constituent	Case Depth—Millimeters Hyper.	Eutect.	Hypo.	Total
Check	None	0.38	0.39	0.86	1.63
Ferroboron	2	0.38	0.33	0.91	1.62
	5	0.32	0.38	0.97	1.67
	10	0.00	0.33	1.15	1.48
Silicon	2	0.36	0.36	0.97	1.69
	5	0.25	0.32	0.83	1.40
	10	0.00	0.31	1.07	1.38
Ferrosilicon	2	0.27	0.36	0.91	1.54
	5	0.00	0.35	0.96	1.31
	10	0.00	0.25	0.99	1.24
	15	0.00	0.12	1.14	1.26
	20	0.00	0.00	1.07	1.07
Magnesium-silicon	5	0.37	0.32	0.90	1.59
	10	0.00	0.27	1.03	1.30
	15	0.00	0.15	0.95	1.10

means of a micrometer ocular, previously standardized for a given objective with a stage micrometer.

The results of these treatments are summarized in Table I, in which are given the nature of the various mixtures and the depth of the various zones of the cases produced.

The results summarized in the table show that all of the four added materials serve to reduce the velocity of carbon absorption, reducing the case depth and eliminating the zone of free cementite. Within the limits of the experimental conditions, the following compositions are found to eliminate the hypereutectoid zone of the case: Ferroboron 10, silicon 10, ferrosilicon 5, magnesium-silicon 10. With closer subdivisions in the composition of the mixtures, some larger variations would no doubt be noted.

Comparison of the results here given for ferrosilicon with those reported in the paper by Mahin and Spencer[1] will indicate that we now find considerably lower proportions of this alloy to be required for the desired effect. This is attributed to the finer state of subdivision of the alloy than was formerly employed, as it will be shown later that activity increases as particle size is diminished.

Photomicrographs illustrating the character of case produced by the various treatments except those in which ferrosilicon was used, are shown in Figs. 1 to 8, inclusive. All specimens were etched in sodium picrate solution, as this method served somewhat better than by using nital, for revealing small quantities of cementite.

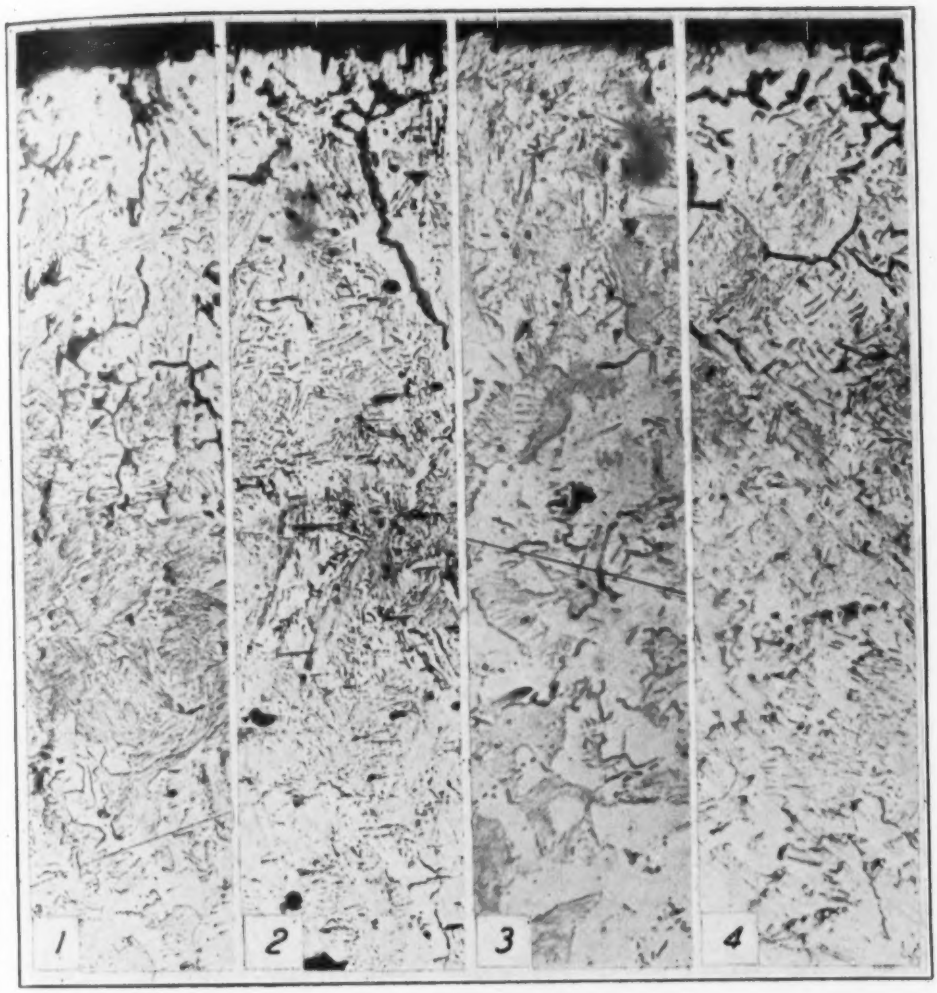


Fig. 1—Photomicrograph of Iron Carburized Without Additions to Carburizer. Etchant Sodium Picrate. × 100.

Fig. 2—Armco Iron, 5 Per Cent of Ferroboron Added to Carburizer. Etchant Sodium Picrate. × 100.

Fig. 3—Armco Iron, 10 Per Cent of Ferroboron Added to Carburizer. Etchant, Sodium Picrate. × 100.

Fig. 4—Armco Iron, 2 Per Cent of Silicon Added to Carburizer. Etchant, Sodium Picrate. × 100.

Action of Carbon Monoxide Upon Ferrosilicon and Upon Silicon, and Carburization of Armco Iron Turnings

The restraining action of silicon, silicon alloys and ferroboron upon the carburizing reactions might, conceivably, be due to any one or more of three causes:

(1) Reduction in the concentration of carbon monoxide within the pot by chemical reduction of this gas, the silicon or boron (or in one case magnesium) acting as the reducing agent.

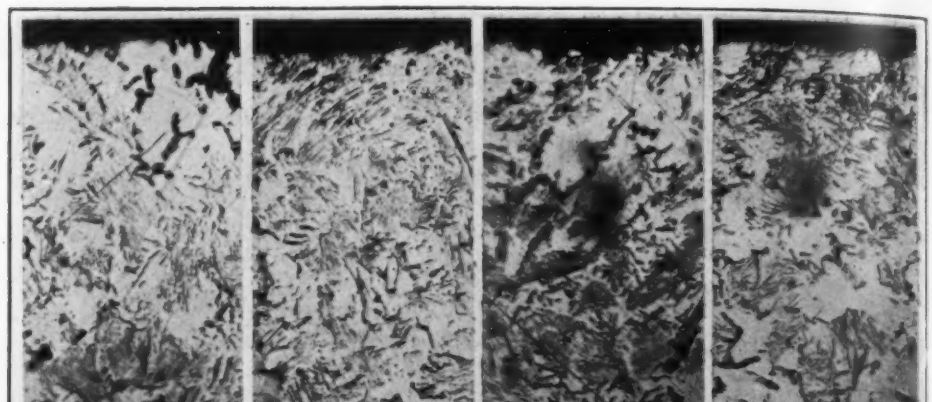


Fig. 5—Photomicrograph of Armco Iron, 5 Per Cent of Silicon Added to Carburizer. Etchant, Sodium Picrate. × 100.
Fig. 6—Armco Iron, 10 Per Cent of Silicon Added to Carburizer. Etchant, Sodium Picrate. × 100.
Fig. 7—Armco Iron, 10 Per Cent of Magnesium-Silicon Added to Carburizer. Etchant, Sodium Picrate. × 100.
Fig. 8—Armco Iron, 15 Per Cent of Magnesium-Silicon Added to Carburizer. Etchant, Sodium Picrate. × 100.

(2) Production of a volatile silicon or boron compound which might act as a negative catalyst.

(3) Production of a volatile silicon or boron compound which would act as a carrier of these elements to the ferrous specimen, the elements so dissolved in, or deposited upon, the surface layers then reducing the rate of solution of carbon. The third theory was favored in the earlier work.

Since pure carbon monoxide is not readily obtained in cylinders,

it was necessary to prepare this gas as needed. The standard methods for preparing the gas for use in small quantities, from formic or oxalic acids, were not suitable in this case but by the method described below the gas could readily be produced in any desired quantities and supplied at whatever rate was required.

The apparatus was set up as shown in Fig. 9. A Coors porcelain tube, with one end closed, was packed with a special coal tar carbon, which had previously been heated, in the process of manufacture, to 2190 degrees Fahr. (1200 degrees Cent.). This is a raw material which is sold for the production of graphite. The porcelain tube was 75 cm long and 5.5 cm internal diameter. Through a rubber stopper in the open end of the tube, was inserted a transparent quartz tube, 3.5 mm internal diameter, extending to the back of the porcelain tube. This quartz tube served to deliver oxygen from a cylinder and exit gases were taken out through a short glass tube which extended barely through the stopper. By this means, practically pure oxygen was delivered at the hottest part of the column of carbon and the mixture of carbon monoxide and dioxide there formed traversed the entire column before being taken out for use.

The entire assembly was placed in an electrically heated muffle furnace, passing through a special muffle plug of fire clay in such a manner as to leave the end bearing the rubber stopper outside the furnace, where it could be kept cool. The furnace was maintained at 1830 degrees Fahr. (1000 degrees Cent.) by means of a pyrometer control.

According to Rhead and Wheeler[14] the equilibrium mixture of carbon monoxide and carbon dioxide, at this temperature and in presence of carbon, contains 99.41 per cent of carbon monoxide and 0.59 per cent of carbon dioxide. This small amount of the dioxide is easily absorbed by passing the gases through sodium hydroxide solution.

As shown in Fig. 9, the exit gases were passed first through two bottles of 15-per cent sodium hydroxide solution, then through a bottle containing basic pyrogallol solution, the latter to remove any small amounts of oxygen from entrapped air, and finally through a drying tower containing calcium chloride and a few sticks of sodium hydroxide. It was considered that gas so prepared and treated would be practically pure carbon monoxide and this is still thought to be the case, with one exception which will be noted later.

[14]Journal of the Chemical Society, Vol. 97, 1910, p. 2179.

A silica tube, 2.7 centimeter internal diameter, was placed in an electrically heated tube furnace, such as is used for the determination of carbon in steel. As it was not practicable to insert a thermocouple during a run, the furnace was first approximately standardized with various resistances in the rheostat and the required rheostat setting for maintaining a given temperature was so determined. Since the

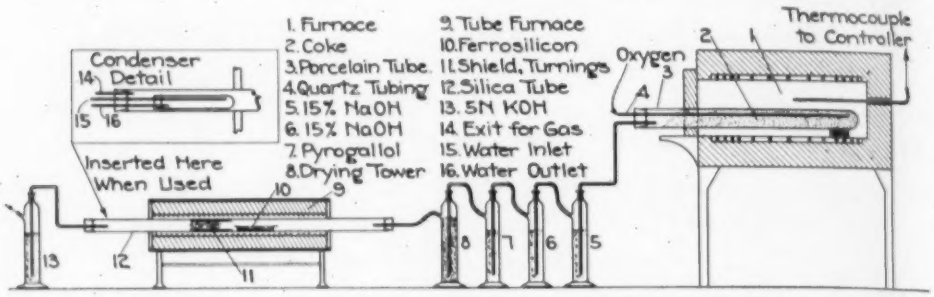


Fig. 9—Apparatus for Determining the Action of Carbon Monoxide Upon Ferrosilicon.

line voltage was subject to some fluctuation, this could not be regarded as a very accurate temperature measurement, but it served for the purpose.

In the silica tube was placed, first, an alundum boat containing ferrosilicon, previously weighed, and this was followed by an open alundum thimble, nearly as large as the tube, in which was placed a quantity of fine turnings of Armco iron. Asbestos twine was wrapped around the thimble to make a fairly close fit in the silica tube. By this means the gases were forced to pass through the mass of turnings, which provided a large relative surface for absorption of the gases.

This tube assembly was placed in the cold furnace and carbon monoxide was passed through for 15 minutes before turning on the furnace heating current. This served to displace the contained air and the furnace then came up to the temperature in a stream of carbon monoxide. After a run was finished the stream of gas was kept running until the furnace had cooled to room temperature.

In some of the experiments the condenser shown in the inset in Fig. 9 was attached at the end of the tube. This served to reveal the presence of any volatile matter which might be liquid or solid at ordinary temperatures. Cold water was circulated through this condenser, when used.

After each experiment, the boat of ferrosilicon was weighed in

order to determine any change in weight. In all cases the weight had increased. The original, untreated ferrosilicon, as well as the contents of the boat after the completion of each experiment, were analyzed to determine the per cents of silicon, iron and carbon. Iron determinations were made by the dichromate method, preceded by fusion in a porcelain crucible with sodium carbonate and potassium nitrate. Decompositions for silicon determinations were made by means of the same fusion mixture, but a nickel crucible was used for the purpose. Carbon determinations were made by direct combustion, followed by absorption in barium hydroxide solution and titration of the absorbent before and after absorption. Silicon determinations were made also upon the fine turnings of Armco iron as used in the tube, as well as upon the turnings after the treatment as described.

Weight Increase in Ferrosilicon, as Calculated from Carbon Added

It might provisionally be assumed that the increase in weight of ferrosilicon, during the treatment, is due to a reaction like the following:

$$2CO + Si \rightarrow SiO_2 + 2C$$

or, for small amounts of aluminum sometimes found in ferrosilicon:

$$3CO + 2Al \rightarrow Al_2O_3 + 3C$$

In effect, this is a direct addition of carbon monoxide to the ferrosilicon and upon this assumption the theoretical weight increase was calculated from the per cent of carbon, as found by analysis. As seen from an inspection of Table II, this calculated weight increase was, roughly, somewhat more than half the actual increase. This may indicate (1) that the equations given above do not provide a complete expression of the reactions, or (2) that a portion of the carbon produced by these reactions was not deposited on the ferrosilicon but that it was carried away by the gas stream.

In the light of experiments (described later in this paper) involving the action of hydrogen sulphide, it seems practically certain that the discrepancy between actual weight increase and that calculated from the per cent of carbon, upon the basis the equations given above, is due to the absorption of sulphur by the iron of ferrosilicon, according to the equation:

$$Fe + H_2S \rightarrow FeS + H_2$$

Table II

Action of Carbon Monoxide upon Ferrosilicon, and of the Siliconized Gases upon Armco "Wool"

Run No.	Time Hours	Temperature °F.	Temperature °C.	Weight Increase of Ferrosilicon Lbs. %	Weight Increase of Ferrosilicon Calc. %	Composition of Ferrosilicon, % Si	Composition of Ferrosilicon, % Fe	Composition of Ferrosilicon, % Si:Fe	C	Silicon in Armco Wool, %
5	2	1740	950	1.65	1.01	....	....	.....	0.82	.....
3	5	1740	950	3.04	2.46	48.1	44.9	1.071	1.41	0.039
6	30	1740	950	6.38	3.66	46.8	46.2	1.015	1.83	0.012
10	70	1740	950	8.20	4.53	44.7	44.5	1.004	2.15	.....
8	15	1920 –1960	1050 –1075	4.27	2.40	48.1	46.7	1.030	1.37	0.012
9	70	1920 –1960	1050 –1075	9.22	3.54	45.6	44.9	1.015	1.76	0.050
		Ferrosilicon as received				49.4	48.5	1.018	0.40	
		Armco iron as received								0.013

The following additional comment may be made in connection with the results as summarized in Table II:

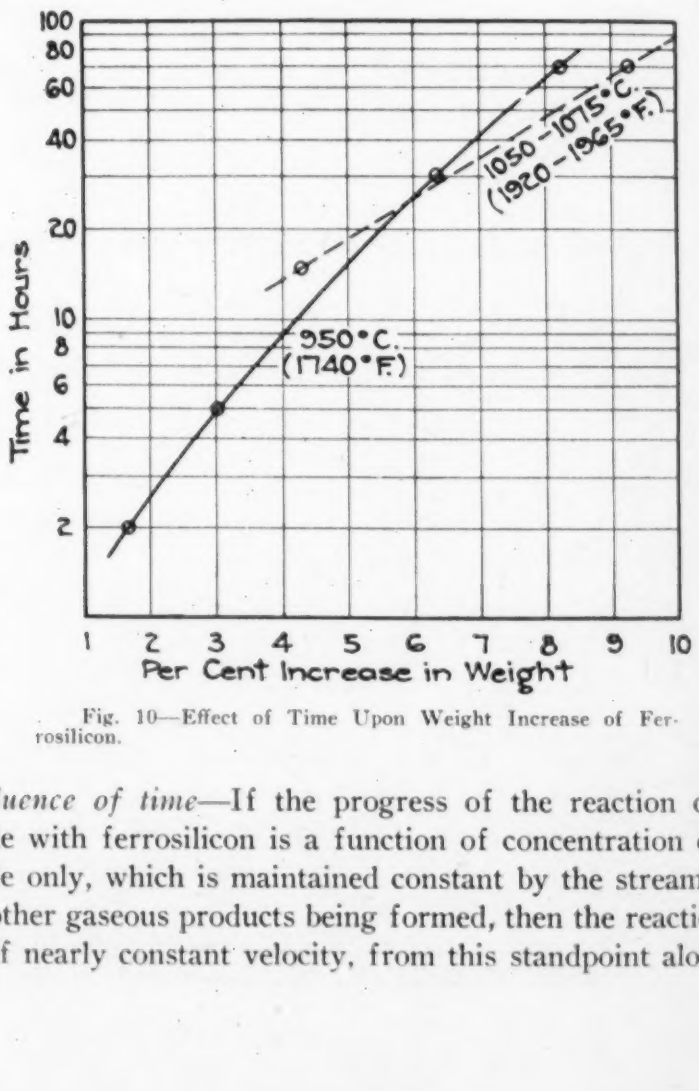


Fig. 10—Effect of Time Upon Weight Increase of Ferrosilicon.

Influence of time—If the progress of the reaction of carbon monoxide with ferrosilicon is a function of concentration of carbon monoxide only, which is maintained constant by the stream of fresh gas, no other gaseous products being formed, then the reaction should be one of nearly constant velocity, from this standpoint alone. Sur-

face changes on the particles of ferrosilicon would be expected to reduce the velocity of the reactions and this effect would increase with time of exposure. Because of the impossibility of determining the quantitative effect of surface changes in the solid, any equation representing the progress of the reaction must be more or less empirical. However, the following equation is found approximately to express the relations existing between time and weight increase:

$$\log \Theta = mW + k,$$

where Θ = time
m = slope
W = per cent weight increase
and k = a constant

The two factors, *time* and *weight increase*, are plotted in Fig. 10, on a semi-logarithmic scale. It is interesting to note that the graphs are nearly straight lines, although this statement has no particular meaning in the case of the experiments at the higher temperature, since only two points were determined.

The Silicon:Iron Ratio—The determination of iron and of silicon in the ferrosilicon, before and after treatment, was made in the hope that this might serve to indicate any loss of silicon as volatile compounds. It is believed, however, that such compounds are formed only in small quantities, which means that they are extremely active in affecting the process of carburization. The figures for the silicon: iron ratio, as shown in Table II, do show some variation but they are not consistent, as might possibly be expected on account of the relatively small losses of silicon and the variability in the composition of commercial ferrosilicon. Small reductions in the ratio were noted in runs (6), (9) and (10), but the possible significance of these is destroyed by increases noted in runs (3) and (8).

Sulphur in the Carburizing Gases—In some of the experiments described above, a bottle of 5N potassium hydroxide solution was connected at the end of the silica tube which was heated by the tube furnace. This is shown as (13) of Fig. 9. The original purpose of this solution was to prevent the backward diffusion of air or carbon dioxide but when the bottle was opened, at the completion of a run, a strong odor of hydrogen sulphide was noticed. A similar odor was observed as coming from the basic solutions immediately following the carbon monoxide generator. Neutralization of the solution in bottle (13), following the tube furnace, by means of hydrochloric acid gave a white precipitate of colloidal sulphur, proving

that hydrogen sulphide had passed through all of the absorbents, as far as the tube furnace.

At the end of a run, 50 cubic centimeters of the solution in the last bottle was removed by means of a pipette, also 50 cubic centimeters of another portion of the solution which had not been in the train during a run was measured. Both of these solutions were neutralized with hydrochloric acid and evaporated for a determination of silica. The unused (blank) solution was found to contain 12.8 milligrams of silica per liter of solution, while the solution which had been used as an absorbent contained 21.4 milligrams per liter.

In nearly all of the experiments a brown deposit was noticed as having collected on the interior of the cooler portion of the silica tube, at the exit end. Some of this was removed and ignited in air, when it burned to a pure white residue, proved to be silica by volatilization with hydrofluoric acid. In some of the runs, made as above, the bottle (13) of potassium hydroxide solution was removed and replaced by the condenser assembly, shown in the inset of Fig. 9. A light yellow to brown deposit was formed also on this and this deposit responded to the same tests as those described for the deposit on the inside of the silica tube. It was noticed also that a portion of the deposit, left on the condenser and exposed to moist air, turned white after a time, leaving a residue of silica.

A consideration of all of these facts suggested the idea that the deposit was composed of one or more of the sulphides of silicon and it is the belief of the authors that such sulphides constitute one (perhaps the only) carrying agency for silicon to the steel or iron being carburized, whether the operation be carried out with solid carburizer or by means of carbon monoxide generated in the manner employed in these experiments. The origin of the sulphur is, of course, the organic sulphur present in the organic matter from which the solid carburizer (or coal tar coke) was made. This sulphur forms hydrogen sulphide by reacting with water vapor during the heating for carburization and it then reacts with ferrosilicon, when this is present, to form silicon sulphide as one of the products.

Two sulphides of silicon are known and their properties have been studied. These are the monosulphide, SiS, and the disulphide, SiS_2. The disulphide is best known. It is formed when silicon is heated in the vapor of sulphur or in hydrogen sulphide and it forms a stable vapor at high temperatures, in the absence of oxygen or water vapor. The pure compound is white, but the product obtained

as above is generally colored by the presence of elementary silicon. The disulphide is known to hydrolyze readily in moist air, hydrogen sulphide and silicon dioxide being formed. These properties are in accord with those determined for the deposit found in the cooler end of the silica tube and on the condenser, in the experiments already described.

Silicon monosulphide is formed in the manner described for the disulphide, but at lower temperatures. At higher temperatures decomposition takes place, presumably as follows:

$$2SiS \rightarrow SiS_2 + Si$$

This again explains the dark color of the deposit, which is probably a mixture of the disulphide and amorphous silicon.

The presence of traces of hydrogen sulphide in the gases evolved from solid carburizers has been noted by Hillman[15], who worked with five commercial mixtures sold as carburizers. The presence of sulphur in coal tar is readily understood and the temperature at which the coke was formed would not be sufficiently high to expel this sulphur completely, except after very long treatments.

In order to obtain further light upon these points, the following experiments were performed[16]:

> A generator for carbon monoxide was prepared as already described. The gases from this generator were passed through a basic solution of cadmium chloride, such as is ordinarily used for the determination of sulphur in steel. A yellow precipitate of cadmium sulphide was obtained in the bottle. It was difficult to see how hydrogen sulphide could have escaped absorption in the two bottles of sodium hydroxide and one of basic pyrogallol of the original train, and it was thought possible that failure to narrow the ends of the delivery tubes in the absorbing bottles was responsible, because of the delivery of large bubbles of gas to the solution.
>
> For the next experiment, all of these tubes were drawn down to the form of "pipette" tips and a train was set up as follows:
>
> (1) Carbon monoxide generator; (2), (3) 15 per cent

[15] *Iron Age*, Vol. 114, 1924, p. 611.

[16] This portion of the work was performed by A. J. Boyle, working with the senior author. Mr. Boyle is a candidate for the degree of Doctor of Philosophy in Metallurgy, in the University of Notre Dame.

sodium hydroxide solutions; (4) basic pyrogallol; (5), (6), (7), (8), (9) ammoniacal cadmium chloride solution, in five absorbing bottles; (10) dilute sulphuric acid, to absorb ammonia derived from the cadmium chloride solutions; (11) calcium chloride, to dry the gases; (12) silica tube in the tube furnace, this containing a boat of ferrosilicon and having the condenser attached at the exit end; (13) ammoniacal solution of cadmium chloride.

During the course of a five-hour run, with the carbon monoxide generator at 1830 degrees Fahr. (1000 degrees Cent.) and the tube furnace at approximately 1740 degrees Fahr. (950 degrees Cent.), a yellow precipitate of cadmium sulphide formed in all of the five bottles of cadmium sulphide solution preceding the tube furnace. This precipitate was quite heavy in the first bottle, somewhat less so in the second and it appeared, in a general way, to diminish more or less regularly in succeeding bottles, the precipitate being negligible in the last bottle preceding the tube furnace. There was no observable precipitate in the cadmium chloride solution following the tube furnace.

The cadmium sulphide in these bottles was titrated by iodine solution, giving a total of 37.06 milligrams of sulphur. This, of course, is merely the sulphur of hydrogen sulphide that has escaped absorption in the three bottles of basic solution, and it must therefore be a minor part of the total sulphur coming from the generator. Among other things, this experiment indicates that absorption of hydrogen sulphide by solutions of either strong bases or alkaline cadmium chloride is far from complete, if the gas is present in more than traces in the gas stream passing through.

Examination of the condenser at the end of the silica tube, after the furnace had cooled, showed only a trace of silicon sulphide and there was no appreciable deposit on the interior of the exit end of the tube itself. Still more significant, in the light of the present investigation, were the results of the next tests.

Two samples of ferrosilicon were tested for the presence of sulphides: (1) The material that had been used in the experiment just described, where the ferrosilicon had been heated in carbon monoxide which had been freed from hydrogen

sulphide, and (2) ferrosilicon used in the earlier experiments, where hydrogen sulphide had passed through the basic solutions to the tube furnace. Small quantities of these materials were placed in separate test tubes and treated with dilute hydrochloric acid solution. Even without warming, sample (2) gave a strong odor of hydrogen sulphide and responded to the basic lead acetate test for this gas. Sample (1) gave no odor of hydrogen sulphide and a negative lead acetate test.

As a result of these experiments, and in the light of Hillman's statements, it seems reasonable to conclude that hydrogen sulphide is present in practically all ordinary carburizing processes and that ferrosilicon, mixed with solid carburizers or present in a stream of carbon monoxide, removes the hydrogen sulphide partly or entirely, with formation of ferrous sulphide and some silicon disulphide, the latter partly volatile.

If the removal of hydrogen sulphide from the carburizing gases is the action which is the basis for the regulating influence of ferrosilicon during carburization, then it must be concluded that hydrogen sulphide possesses an importance in carburizing processes which has heretofore been overlooked—namely, that in small quantities it promotes carburization and that if it is removed from such possibility of influence, the rate of carbon absorption by iron or steel is thereby reduced. Possibly this may explain why pure carbon monoxide is a slow carburizer, even when the carbon dioxide produced by the reaction

$$Fe + 2CO \rightarrow Fe, C \text{ (solid solution)} + CO_2$$

is swept away by the gas stream of fresh carbon monoxide.

The following reversible reaction, at high temperatures, is postulated as an explanation of the positive influence of hydrogen sulphide upon the velocity of carburization:

$$H_2S + 3CO \rightleftarrows H_2O + SO_2 + 3C$$
$$(\text{or } H_2S + CO \rightleftarrows H_2O + S + C)$$

If the gases are in equilibrium with only small concentrations of water vapor and sulphur dioxide in presence of an excess of carbon, then at the surface of a piece of steel which is being carburized the carbon is absorbed and the reaction proceeds as above, from left to right. The mixture of water vapor and sulphur dioxide then, by dif-

fusion, comes into contact with the excess of carbon of the carburizer and the reaction is reversed:

$$H_2O + SO_2 + 3C \rightarrow H_2S + 3CO$$
$$(\text{or } H_2O + S + C \rightleftarrows H_2S + CO)$$

Hydrogen sulphide would then, upon this assumption, act as a positive catalyst for the carburizing reactions.

Experimental work is under way, designed to submit this hypothesis to a quantitative test but at the date of submission of manuscript for this paper, they are not complete and a report will be deferred to a later date.

Silicon Absorption by Armco "Wool."—An increase in the silicon content of the Armco iron turnings upon treatment with carbon monoxide which had been passed over heated ferrosilicon, occurred in three of the five runs in which an analysis was made, as noted in Table II. It may also be stated here that in nearly all of the experiments recorded in this report, as well as in other experiments to be reported later, analyses were made of treated turnings or of thin layers turned from the surfaces of cylindrical specimens carburized in the presence of ferrosilicon. With few exceptions, these analyses showed that the silicon content of the turnings had been increased. In some cases the increase was small, but unmistakable. In a very few, no increase in silicon content was detected. The general rule is then in accord with that noted by Mahin and Spencer.

It seems likely that the exceptions noted in Table II, for runs (6) and (8), may be connected with certain irregularities in the conduct of the experiments, as follows:

The generation of carbon monoxide involves consumption of the coke contained in the generator and the latter was, therefore, occasionally opened between successive experiments, for the insertion of fresh coke. It is also known that the brown deposit was not always formed at the exit end of the silica tube. At the time the experiments were being conducted, no attention had been given to the possibility of the formation of silicon sulphide, or to the possible influence of this compound upon the progress of carburization. There may be some connection between these various facts. If the continued heating of the coke in the generator finally exhausted its sulphur content, or nearly so, hydrogen sulphide formation, and

therefore silicon sulphide formation, would drop nearly to zero until fresh coke was added. And, again, if silicon sulphide is the carrying agent for silicon in experiments where this compound was lacking there would be no conductance of silicon to iron turnings. As we have no record of correlation of these points, the reasoning must remain questionable until further experimental results are available.

It has already been shown that ferroboron influences the progress of carburization in a manner similar to that shown by ferrosilicon and elementary silicon. It is interesting to note that boron sulphide, B_2S_3, is formed in a manner analogous to that of silicon sulphide—by heating boron in the vapor of sulphur or in hydrogen sulphide gas. There seems every reason for supposing that the ferro-alloys of these elements should react in the same way, since each alloy must contain a considerable excess of the elementary phase, silicon or boron.

Action of Carbon Monoxide upon Elementary Silicon—Silicon influences carburization in the same manner as does ferrosilicon and the experiment just described and discussed was therefore repeated, elementary silicon of a technical grade being substituted for the ferrosilicon in the boat. Otherwise there was no variation in the conduct of the experiment. Data obtained for a run of 50 hours at 1740 degrees Fahr. (950 degrees Cent.) are as follows:

Composition of Silicon—Per Cent						Weight Increase of Silicon—Per Cent
As Received			After Run			
Si	Fe	C	Si	Fe	C	
95.8	1.18	<0.01	94.0	1.11	0.29	2.44

The weight increase of the silicon is considerably greater than may be accounted for by the small increase in carbon per cent, upon the assumption of simple addition of carbon monoxide according to the equation:

$$Si + 2CO \rightarrow SiO_2 + C$$

It may therefore be concluded that there was some oxidation of silicon by some other means, or that amorphous carbon so produced, as above, was carried away by the stream of gas. The weight increase noted is then a net increase, loss of carbon and of silicon sulphide being included in this figure.

Duration of Effectiveness of Ferrosilicon—If the influence of ferrosilicon upon the progress of carburization were to diminish with

Table III

Effect of Previous Use upon the Effectiveness of Ferrosilicon at 1740 Degrees Fahr. (950 Degrees Cent.)

Metal Carburized	Time Hours	Ferrosilicon Previous Treatment with CO	Per Cent in Mixture	Case Depth, mm. Hyper.	Eut.	Hypo.	Total
Ingot Iron	5	60 hours at 950° C. 1740° F.	15	0.00	0.56	0.88	1.44
	9	60 hours at 950° C. 1740° F.	20	trace	0.54	1.31	1.85
Steel	5	None	20	0.00	0.64	....	....
	5	5 hours at 950° C. 1740° F.	20	0.00	0.63	....	....
	5	60 hours at 950° C. 1740° F.	20	0.00	0.86	....	....

continued use, due to the volatilization of some impurity contained in small amounts, or to permanent alteration of surface conditions on the particle, this fact would have an important bearing upon the continued usefulness of the material as a constituent of any commercial carburizing mixture. In order to determine this point mixtures were made, in which the ferrosilicon used had previously been treated with carbon monoxide for varying periods, according to the method already described. Both 15 and 20 per cent mixtures of this with carburizer were made and these mixtures were used to carburize both Armco ingot iron and steel containing 0.67 per cent of carbon. This relatively high carbon steel was used in order to make possible the building up of a high carbon case in a comparatively short time.

The results of this experiment are recorded in Table III. For the steel, total case depths are not given, on account of the difficulty involved in determining the inner boundary of the hypoeutectoid zone of the case itself.

Carburization for five hours at 1740 degrees Fahr. (950 degrees Cent.) has repeatedly been shown to give a decidedly hypereutectoid outer case, when no ferrosilicon is used in the mixture, and 20 per cent of ferrosilicon (or even 5 per cent, when finely pulverized) has been found to eliminate this zone. From the results shown in Table III it is evident that even ferrosilicon which had been heated for 60 hours in a stream of carbon monoxide retains its effectiveness in this respect. If any coating of carbon is formed on the particles, such as might reduce the effectiveness of contact with carburizing gases, this carbon is evidently detached in the ordinary process of handling the powdered material.

Figs. 11, 12, 13 and 14 illustrate the appearance of the transverse

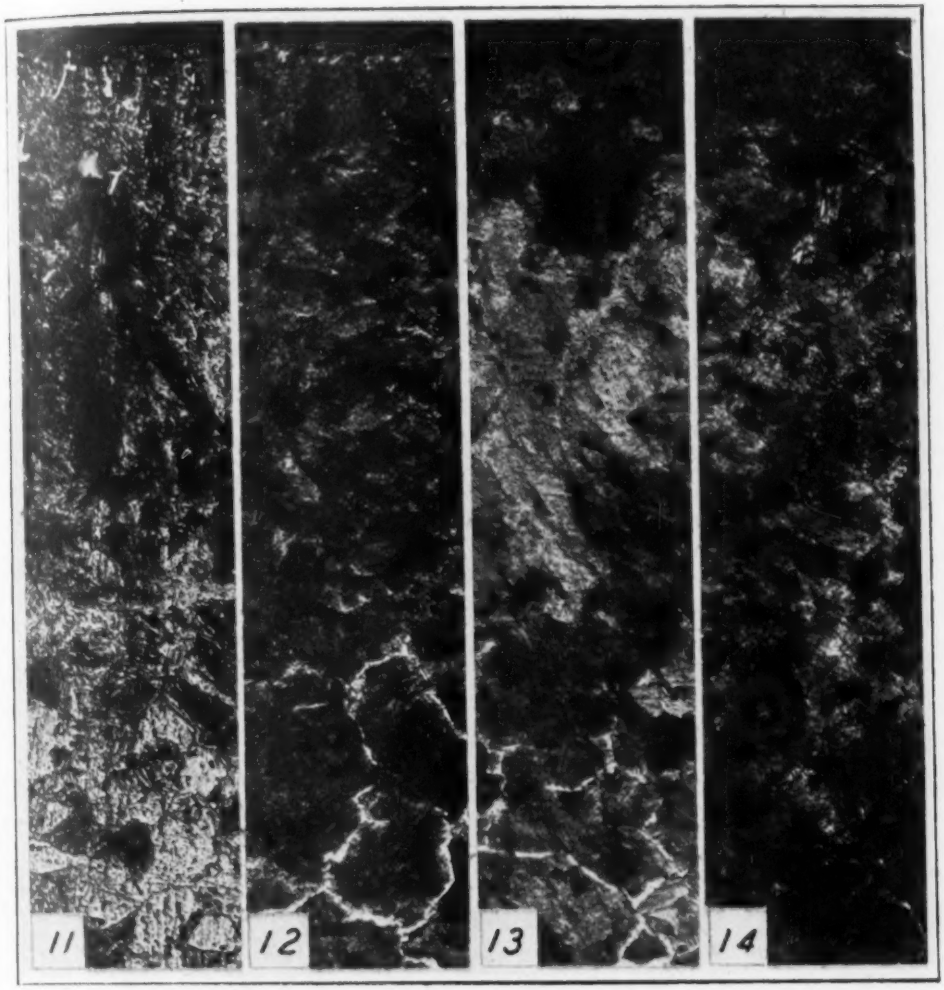


Fig. 11—Photomicrograph of Armco Iron. Carburizer Mixed with 20 Per Cent of Ferrosilicon which had been Heated with CO for 60 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Carburized 9 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Etchant, Nital. × 100.

Fig. 12—Steel, 0.67 Per Cent Carbon. Carburizer Mixed with 20 Per Cent of Fresh, Untreated Ferrosilicon. Carburized 5 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Etchant, Nital. × 100.

Fig. 13—Steel, 0.67 Per Cent Carbon. Carburizer Mixed with 20 Per Cent of Ferrosilicon which had been Heated with CO for 5 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Carburized for 5 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Etchant, Nital. × 100.

Fig. 14—Steel, 0.67 Per Cent Carbon. Carburizer Mixed with 20 Per Cent of Ferrosilicon which had been Heated with CO for 60 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Carburized for 5 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Etchant, Nital. × 100.

sections of the carburized pieces. Here, as well as in Table III, it is seen that the ferrosilicon which had previously been exposed to carbon monoxide for 60 hours permitted the formation of a somewhat deeper eutectoid case in the steel than that treated for a shorter

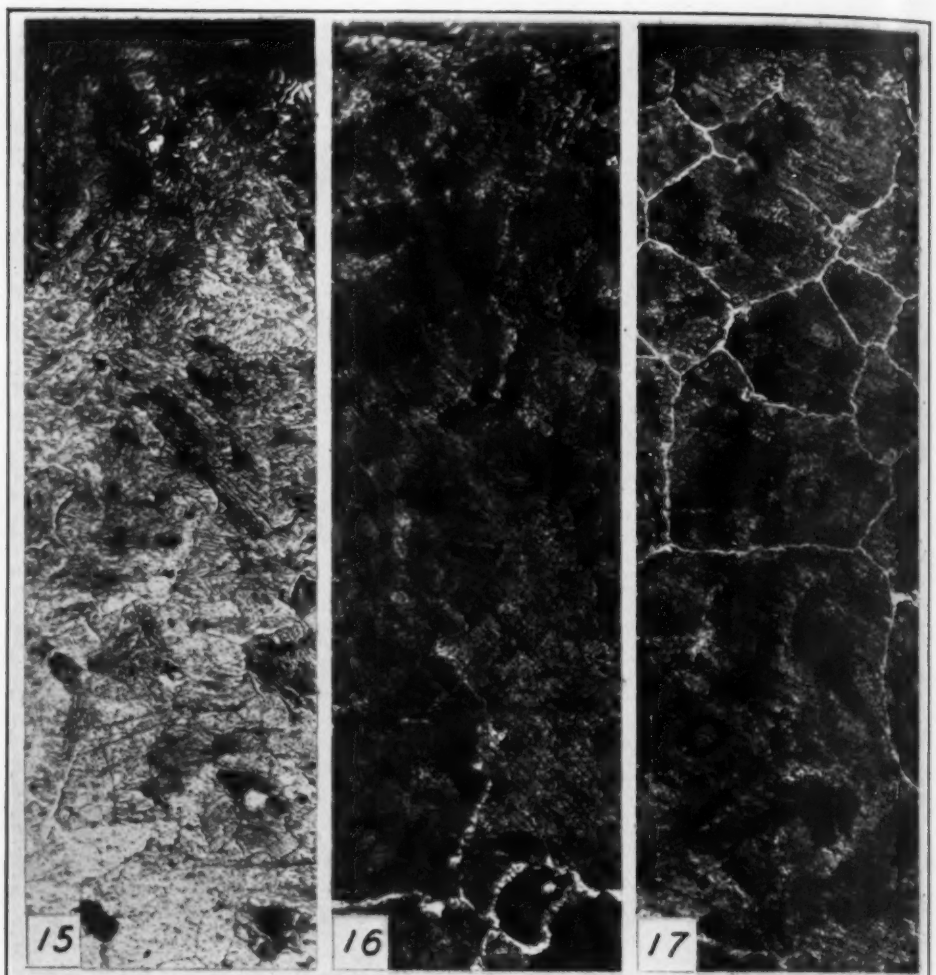


Fig. 15—Photomicrograph of Armco Iron. Carburizer Mixed with 5 Per Cent of Powdered Aluminum. Carburized for 9 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Etchant, Nital. × 100.

Fig. 16—Steel, 0.67 Per Cent Carbon. Carburizer Mixed with 5 Per Cent of Powdered Aluminum. Carburized for 5 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Etchant, Nital. × 100.

Fig. 17—Steel, 0.67 Per Cent Carbon. Carburizer Mixed with 5 Per Cent of Powdered Magnesium. Carburized for 5 Hours at 950 Degrees Cent. (1740 Degrees Fahr.). Etchant, Nital. × 100.

time, or not treated at all. But even in this, there is no free cementite in the outer layers.

Chemical Reduction of Carbon Monoxide by Ferrosilicon as a Possible Cause of Action—If actual destruction of carbon monoxide by the formation of silicon dioxide and free carbon were the principal (or the only) cause of the influence of ferrosilicon upon carburization, then it should be true that other active reducing agents should

Table IV

Influence of Aluminum and Magnesium upon Carburization at 1740 Degrees Fahr. (950 Degrees Cent.)

Metal Carburized	Time Hours	Carburizing Mixture	Case Depth, mm Hyper.	Eut.	Hypo.	Total
Ingot iron	5	With 5% Al	0.00	0.51	0.97	1.48
	5	With 10% Mg	0.29	0.41	0.71	1.41
	9	With 5% Al	0.00	0.59	1.31	1.90
Steel, 0.67% C.	5	With 5% Al	0.00	0.65	....	....
	5	With 5% Mg	0.76	0.65	....	....

show similar effects. Accordingly, mixtures were made of the carburizer with powdered aluminum and magnesium, respectively. The heats of oxidation of these elements, as well as that of silicon, are all much higher than that of carbon monoxide and they might be expected to reduce the gas. Silicon in the form of ferrosilicon would naturally be less active than the elementary metals.

The results of the carburization by mixtures with aluminum and magnesium are summarized in Table IV. Photomicrographs illustrating the character of case obtained when aluminum was used are shown in Figs. 15 and 16, and that when magnesium was used, in Fig. 17. It will be seen that magnesium is not particularly effective, notwithstanding its well known activity as a reducing agent.

Aluminum reduces the case depth very decidedly and it eliminates the zone of free cementite entirely, even when only 5 per cent is used in the mixture. It should be remembered, however, that aluminum possesses a comparatively high vapor pressure at the carburizing temperature, and the vapor undoubtedly forms a high-aluminum alloy coating on the steel or iron, as is the case in the process of calorizing. This coating inhibits the absorption of carbon by the iron or steel and the total effect is therefore a combination of this effect with the possible reduction of concentration of carbon monoxide within the carburizing chamber.

Carburization of High-Carbon Steels—It was thought desirable to apply every possible test to determine whether the restraining effect of ferrosilicon may be due, in any measure, to reduction of the concentration of carbon monoxide within the pot by chemical decomposition, according to the equation already given. The disappearance of this gas, through the formation of two solids, would result in the drawing in of fresh air, the oxygen of which would ultimately form more carbon monoxide, a portion of which again would be de-

composed. The ultimate result of this would be a continued dilution of the carburizing gases by unused nitrogen of the air and this is, of course, the action during any normal carburizing process when using solid carburizer.

If dilution from this source is appreciably greater when ferrosilicon (or any of the other restrainers) is mixed with the carburizer, then these special mixtures should not only fail to carburize high carbon steels—they should actually cause decarburization.

Six carbon steels were selected, varying in carbon content from 0.54 to 1.40 per cent. Two pieces from each steel were cut and cleaned on the lathe for carburization. One piece of each steel was packed in unmixed carburizer, as a check piece, the other being packed in carburizer mixed with 20 per cent of ground ferrosilicon. In each case, the two pieces were placed in the same furnace and carburized at 1740 degrees Fahr. (950 degrees Cent.) for 5 hours, while a second set of the specimens was carburized in the mixture, at the same temperature but for 10 hours. The results of this experiment are tabulated in Table V.

Table V
Carburization of High Carbon Steels, with and without Ferrosilicon Mixed with Carburizer

Carbon in Original Stock, %	Character of Case		
	In Unmixed Carburizer	In Carburizer with 20% of Ferrosilicon	
	Carburized 5 Hours	Carburized 5 Hours	Carburized 10 Hours
0.54	Hypereutectoid	Eutectoid	Eutectoid
0.67	Hypereutectoid	Eutectoid	Eutectoid
0.83	Hypereutectoid	Eutectoid	Eutectoid
0.87	Hypereutectoid	No change	Slightly Hypereutectoid
1.1	No change	No change	No change
1.4	No change	No change	No change

As shown in the table, the special carburizer failed to build up a hypereutectoid case in any of the steels except the one which was eutectoid in the beginning, and this only after a treatment of 10 hours. Even after this treatment the case was eutectoid to the edge, over about one-third of the circumference.

There was no decarburization of the high carbon steels, so far as could be detected by microscopic examination, from which it may safely be inferred that excessive dilution of carburizing gases by nitrogen has exerted no important influence upon the effective concentration of carbon monoxide, in experiments with special mixtures with ferrosilicon.

Silicon Additions to Case—It has repeatedly been proved that when iron or steel is heated in direct contact with ferrosilicon, silicon is imparted to the ferrous specimen by the process of solution (or by deposition and partial solution). In simple mixtures of carburizer and ferrosilicon this offers some difficulty in the matter of chemical analysis of surface layers to determine silicon absorption by gaseous transfer, as there is always a question as to what proportion of the silicon so found may have been imparted to the metal by solid contact.

A special carburizer was prepared for experimental use by a manufacturer of commercial carburizers. This special material has as its base a mixture of coal tar coke and corn cob charcoal, energized with carbonates of barium and calcium. Granules of the carburizer were coated with powdered ferrosilicon, which was bound to the granule by a coating of coal tar, the composite granule then being charred. By this means, direct solid contact of ferrosilicon with metal being carburized is prevented.

Armco iron rounds were cleaned and smoothed on the lathe and pieces were carburized in this special carburizer, the standard run of 5 hours at 1740 degrees Fahr. (950 degrees Cent.) being made.

In a second experiment similar pieces were carburized for the same temperature and time, but using a mechanical mixture of the commercial carburizer which was used in most of the other experiments, with 20 per cent of ferrosilicon. In this case ferrosilicon was prevented from coming into contact with the iron piece by surrounding the piece by a half-inch layer of carburizer *without* ferrosilicon, held in place by cheese cloth, and then packing the pot with the mixed carburizer. The cheese cloth would of course char during the heating but its usefulness had ended when the pot was packed.

After carburization, the pieces were carefully cleaned and all were normally smooth and bright. They were placed in the lathe and layers 0.003-inch in depth were turned from the surfaces. A silicon determination in all of these, together with a determination made upon the original uncarburized ingot iron, gave the results shown in Table VI.

There is no special degree of agreement in the analyses of layers from the carburized specimens but there is also no reason for expecting such agreement, on account of the impossibility of turning

Table VI
Silicon Additions During Carburization

Specimen No.	Method of Carburizing	Per Cent Silicon
0	Uncarburized (check)	0.013
1	A	0.055
2	A	0.025
3	B	0.040
4	B	0.019

Explanation: "A," method using special carburizer.
"B," method using mixed carburizer.

exactly the same depth of layers from all of the rounds after the slight distortion caused by carburization has taken place. Upon the obvious assumption that there is a silicon gradient even within the layers removed, the actual silicon content of the first few ten-thousandth inch must be considerably higher than that represented in the above analyses.

Effect of Particle Size Upon Activity of Ferrosilicon

In the work reported by Mahin and Spencer[1] the ferrosilicon used was simply ground to pass a 50-mesh sieve, so that it contained all of the smaller sizes, as well as the maximum of that passing the sieve. In some of the earlier work of the present investigation another lot of ferrosilicon was obtained, this having been screened by the manufacturers to pass the 40-mesh sieve and to be retained on the 60-mesh sieve. Mixtures of this with carburizer appeared to be almost identical with unmixed carburizer, so far as the effect upon case depth and characteristics were concerned. It was thought that this might have been due to the absence of small particles, with consequently lower values for exposed surfaces. In the following experiments the ferrosilicon was ground in a ball mill and then screened into five fractions, the particle sizes varying from a maximum of that just passing a 40-mesh sieve to a minimum of that passing a 325-mesh sieve (and smaller). Mixtures of 20 per cent of each of these fractions with 80 per cent of commercial carburizer were made and Armco ingot iron pieces were carburized with the mixtures, all for 5 hours at 1740 degrees Fahr. (950 degrees Cent.). The specimens were weighed before and after the runs, in order to determine the weight of carbon added. Transverse sections were then cut from the pieces and these were polished and etched for case depth measurements. The results are summarized in Table VII, in

Table VII

Effect of Particle Size of Ferrosilicon Upon its Activity as a Restrainer

Mesh of Ferrosilicon	Carbon Added gm./sq. dm.	Case Depth, mm Hyper.	Eut.	Hypo.	Total
40–60	0.766	0.44	0.37	0.93	1.74
Check. No ferrosilicon	0.772	0.38	0.39	0.86	1.63
60–100	0.756	0.41	0.37	0.87	1.65
Check. No ferrosilicon	0.778	0.39	0.37	0.90	1.66
100–200	0.705	0.24	0.54	0.90	1.68
Check. No ferrosilicon	0.779	0.37	0.40	0.85	1.62
200–325	0.527	0.00	0.48	0.62	1.10
Check. No ferrosilicon	0.847	0.52	0.48	0.74	1.74
Below 325	0.469	0.00	0.44	0.64	1.08
Check. No ferrosilicon	0.883	0.50	0.52	0.74	1.76

which the carbon added is stated in grams per square decimeter of exposed surface of the ingot iron.

These figures indicate that as the particle size changes from a maximum of that passing the 40-mesh sieve to that passing the 325-mesh sieve, the influence of the alloy upon carburization increases from a practically negligible value to a reduction of absorbed carbon amounting to 39 per cent, and a reduction of total case depth amounting to 38 per cent, with total elimination of the hypereutectoid zone of the case. It is thus evident that economical use of ferrosilicon for this purpose must involve preliminary grinding and screening of the material and in this case considerably smaller proportions may be used.

Gases Evolved from Ferrosilicon—In the first report upon this work[1] the suggestion was made that the silicon-carrying agency might be hydrides of silicon. This suggestion had not been tested and it was made merely because the odor which is observed as coming from freshly broken pieces of ferrosilicon resembles that which is more or less common to silicon hydride, phosphine, arsine and acetylene.

This idea is no longer entertained by the authors. Silicon hydride is extremely unstable at carburizing temperatures and its existence in more than the merest traces therefore seems impossible. Since the publication of the first report, the literature has been more carefully examined in this connection and it is evident that the evolution of both phosphine, PH_3, and arsine, AsH_3, from moist ferrosilicon has been observed for many years, although no mention of the formation of silicon hydride has been found. In fact, the occurrence of explosions in the holds of vessels carrying shipments of ferrosilicon has been traced to the formation especially of phosphine and this has given rise to regulations concerning the carrying of such

shipments in ocean vessels. A fairly comprehensive discussion of these has recently been published.[17]

Pellew investigated the explosions of casks of 50 to 60 per cent ferrosilicon and found them to be due to the formation of phosphine and arsine.[18]

Dupre and Lloyd found that moisture caused the evolution of phosphine, but not of acetylene or silicon hydride.[19]

The constituent which reacts with moisture for the generation of phosphine is considered by Kurnakoff and Urasoff[20] to be a quaternary unstable system, containing the solid solution of iron, silicon, phosphorus and aluminum. The ternary systems iron-silicon-phosphorus and iron-silicon-aluminum are said not to be affected by water.

Lebeau measured and analyzed the gases evolved from 50 per cent ferrosilicon when it reacts with water.[21] Working with three different lots, he obtained total gases from 47.4 to 375 cubic centimeters per kilogram. In these gases the per cent of hydrogen varied from 1.0 to 56.0; of phosphine from 29.87 to 72.9; and of arsine from 2.43 to 54.87.

Were it not for the fact that ferrosilicon has been demonstrated to retain its restraining effect upon carburization after repeated or prolonged use of the finely divided material, it might be supposed that either phosphorus or arsenic plays some part in the demonstration of this phenomenon. Although this seems extremely unlikely, this phase of the matter will be investigated.

Summary

The restraining influence of ferrosilicon upon the progress of carburization has been rechecked and confirmed. It has been demonstrated also that elementary silicon, magnesium-silicon and ferro-boron produce effects similar to those of ferro-silicon, when mixed in a finely divided condition with solid carburizers. In the instance of the use of ferrosilicon for this purpose, the positive addition of

[17] *Metals and Alloys*, Vol. 1, 1930, p. 373.

[18] *Journal*, Society of Chemical Industry, Vol. 33, 1914, p. 774.

[19] *Journal*, Iron and Steel Institute, Vol. 65, 1904, p. 30.

[20] *Zeitschrift für angewandte Chemie*, Vol. 29, 1916, p. 458.

[21] *Revue de métallurgie, mémoires*, Vol. 6, 1909, p. 907.

silicon to the surface layers of the iron or steel piece has been proved, although the silicon increase calculated for a layer 0.003 inch in depth is relatively small.

It has been established that finely divided ferrosilicon, when heated in a stream of impure carbon monoxide, removes a large portion of the hydrogen sulphide carried by the carbon monoxide, and it may be assumed that when the powdered alloy is mixed with solid carburizers the effect is the same. Increase in activity with decrease in particle size is in harmony with this assumption, since the effect of the alloy is necessarily based upon surface action.

The assumption that hydrogen sulphide acts as a positive catalyst in carburizing processes is in harmony with the following known facts:

1. The weight increase of ferrosilicon, heated in a stream of carbon monoxide, cannot be accounted for by simple absorption of carbon monoxide itself, forming silicon dioxide and carbon, but it may be—at least qualitatively—by a consideration of the fact that sulphur also is known to be absorbed.

2. Ferrosilicon absorbs hydrogen sulphide and it also reduces the velocity of carburization.

3. Pure carbon monoxide is a "slow" carburizer, even when a stream of the gas constantly carries away the carbon dioxide that is formed during carburization.

4. A pure form of carbon, even when energized with carbonates, is a slower carburizer than is carbon from various charcoals and cokes.

The regulating influence of ferrosilicon upon carburizing processes may be due to any one or more of the following demonstrated actions:

1. The formation of volatile silicon disulphide, which carries silicon to the surface of the ferrous piece, the higher-silicon alloy then retarding the absorption of carbon.

2. Reduction of the concentration of carbon monoxide through the formation of silicon dioxide and carbon, thus drawing more air into the pot and ultimately diluting the carbon monoxide by nitrogen.

3. Removal of hydrogen sulphide which, when mixed in small proportions with carburizing gases, acts as a positive catalyst to carbon absorption.

At present, the evidence seems to favor the third action, as ac-

counting in the greatest measure for the effect of ferrosilicon upon the depth and character of case obtained under specified time and temperature conditions.

It has been shown that the ratio of ferrosilicon to total mixed carburizer may be reduced considerably below that of 20 per cent, the value established by Mahin and Spencer, if the alloy be very finely divided, as little as 5 per cent exerting a marked effect, in regulating the character of case.

The removal of hydrogen sulphide from the carburizing gases may possess a significance that has previously been neglected, simply because this gas is present in practically all carburizing processes. If hydrogen sulphide also adds sulphur in appreciable quantities to the surface of the piece that is being carburized, the physical properties of the case should be materially improved by its removal from the possibility of such a contaminating effect. The investigation is still under way and a thorough quantitative test of all of these possibilities will be attempted.

Acknowledgment

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The American Rolling Mill Company, for Armco ingot iron; The Rodman Chemical Company and Char Products Company, for carburizers; The Vanadium Corporation of America, for ferrosilicon and other ferroalloys used in experiments later to be reported; Midvale Steel Company, for high carbon steels; and Mr. Peter C. Reilly, president of the Republic Creosoting Company, for heat treating furnaces generously added to our equipment and indispensable for the experimental work here reported.

DISCUSSION

Written Discussion: By Oscar E. Harder, assistant director, Battelle Memorial Institute, Columbus, Ohio.

Prof. Mahin and Mr. Mootz are to be complimented upon their contribution to the knowledge of the carburizing process.

Before the commercial importance of their proposed process can be evaluated, there are certain additional factors which should be determined.

While a higher carburizing temperature apparently may be used without producing certain undesirable case characteristics, no consideration seems

to have been given to the effect of this higher temperature on the properties of the core. The question may be asked as to whether or not the higher temperature would also cause excessive grain growth of the core.

The Armco ingot iron used in these researches is probably to be classed as an "abnormal" carburizing steel or iron, as found by Harder, Weber, and Jerabek[1] and, therefore, might not show the same changes in case and core characteristics as a more normal carburizing steel. It is difficult to determine from the micrographs presented the normality of the various samples.

The authors do not seem to have determined the sulphur contents of their carburized specimens, and one immediately wonders if both the silicon and the sulphur go into the case.

The authors' observations seem to have an important bearing on carburization by gas and may possibly show some correlation with such research as that reported by Guthrie and Wozasek.[2]

The authors have not reported on the relative rates of diffusion of silicon and boron, and presumably it would be interesting to make this observation.

It would seem unfortunate if manufacturers of carburizing compounds intentionally added a hydrogen sulphide producing material, in order to positively catalyze the carburizing reaction, if by so doing sulphur was added to the steel, thereby producing a high sulphur content in the case.

G. M. Enos:[3] I think it is obvious that this contribution is one of value, especially as it is more of a progress report than a final set of conclusions. I would like to ask the authors about the effect of ferrosilicon when used with a pure carbon base such as sugar carbon, or when used with other carburizers than the one which they mentioned. Specifically, what types of carbon bases have been employed in this investigation and what energizers have been used?

It would be interesting to know if there are any plants using ferrosilicon in the carburizers on a commercial basis. With regard to some of the silicon determinations mentioned in the text, it would be interesting to know whether silicon was actually determined on the cylinders. Dr. Mahin spoke of the determination of silicon, but I do not believe he has specifically stated that the determination was made upon the samples themselves. It would be interesting to know whether the ferrosilicon has an effect on the regenerative characteristics of commercial compounds. I am aware that Dr. Mahin may not be able to answer all of these questions at the present time, but perhaps he may be able to do so at some future date.

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[1]O. E. Harder, L. J. Weber and T. E. Jerabek, "Studies on Normal and Abnormal Carburizing Steels", TRANSACTIONS, American Society for Steel Treating, Vol. 13, p. 961, 1928.

[2]R. G. Guthrie and Dr. Oscar Wozasek, "Gas Carburization of Steel", TRANSACTIONS, American Society for Steel Treating, Vol. 12, p. 853, 1927.

[3]Assistant professor of metallurgy, University of Cincinnati, Cincinnati, Ohio.

Authors' Closure

The question raised by Dr. Harder with regard to the grain size of the core is an important one. The grain structure of the core of any untreated carburized piece is coarse and while a high temperature does promote grain growth we may expect in most cases to refine this grain before the piece is finally finished. I do not think we could say that the coarsening effect of this treatment upon the core is such as to be unusual. As to the abnormal characteristics of Armco iron, that, of course, is what we always notice. The only objection I know of to using Armco iron for experiments of this character is that it is not as easy to determine the depth of the hypereutectoid case in abnormal iron or steel as in normal iron or steel. Armco iron is used in the majority of the work because it is easy to determine the total depth of case. If you use a very low carbon steel, you can usually determine pretty accurately the inner boundaries of the case and with Armco iron this is especially easy.

The sulphur content of the piece before and after treatment has not been determined. I may remark that attention was concentrated upon the matter of sulphur only very late in the work, in fact just before the manuscript had to be submitted in time for preprinting, so that we have, as you can see, a very large amount of work to do still in connection with clearing up the action of hydrogen sulphide. Whether the explanation of the action, as given in the paper, is correct I am not sure, in other words, we do not know a great deal about this action.

I do not know just what Dr. Harder means when he says that it would be unfortunate if manufacturers should add hydrogen sulphide producers to carburizers for the purpose of speeding up carburization. I should not consider that to be advisable, although it is a fact that at least one patent has been obtained for a carburizer in which sulphur compounds are intentionally added for the purpose of increasing the speed of carburization. It seems to me that this procedure has certain disadvantages that are rather obvious, but the fact that the addition of a sulphur compound has at least been said by a patent office to increase the speed of carburization would lend a certain amount of support to the idea that the removal of sulphides from carburizing gases would work in the other direction.

Replying to the discussion of the second speaker (Dr. Enos) as to the effect of ferrosilicon, when used with pure carbon or sugar carbon. We have already completed one or two experiments using sugar carbon but they have not been conclusive and that work is still to be done. Naturally it would occur to one that in the attempt to eliminate all possible variables the simplest method would be to use pure carbon, with and without energizing. We had intended doing both of those things. However, I can make no report upon that now.

Beside the carburizers mentioned on page 4 (made by the Rodman Chemical Company) we have used also carburizers made from a coal-tar coke basis. Specimens have been made for us by the Char Products Company, in which ferrosilicon has been incorporated within the pellet, so that when the piece is packed in the carburizer there is no contact of ferrosili-

con with the ferrous piece. Sodium carbonate energizers cannot be used in these cases because ferrosilicon is decomposed at high temperatures by sodium carbonate. The pellet size has not been varied much, but the particle size of ferrosilicon has been. There are certain tables at the bottom of the present paper in which the results are set forth. We find, of course, that the very finely divided ferrosilicon is the most efficient ferrosilicon and that is responsible for the fact that in the paper published two years ago we arrived at the 20-per cent mixture as the best one, while now with ferrosilicon ground to a 200-mesh or finer size we obtain the total elimination of the hypereutectoid zone with only 5 per cent. This is, of course, rather important when it comes to the matter of expense.

As to the question as to whether any plants now use ferrosilicon, I do not know of any. If anyone does I would be interested in learning of it. We have been chiefly interested in studying the more scientific aspects of the question because before you can commercialize any process you have to know something more about it and you can not learn this from a few experiments.

RELATION OF STRUCTURE TO SURFACE HARDNESS OF A CASE HARDENED STEEL

By H. W. McQuaid and O. W. McMullan

Abstract

This paper gives the results of an investigation made to determine the cause of non-agreement between the file test and other hardness test methods used in checking case hardened work. The results show how the hardness test results may be confusing on case hardened work, particularly where high carbon alloy cases are obtained.

The effect of the condition of the precipitated carbides, the carbon content of the hypereutectoid zone, and the solution of the carbides in the hypereutectoid zone, on the hardness test results is shown.

The results indicate the care required in selecting hardness specifications and testing methods to meet the particular requirements of service. It is shown how it is possible to obtain file hard surfaces which show very low Rockwell and scleroscope hardness and vice versa. The recommendation is made that on case hardened alloy steels that the effect of the specified heat treatment and the requirements of service be well known before a definite range of hardness or method is specified.

THE plant metallurgist is faced continually with the problem of producing case hardened parts to meet arbitrary hardness specifications set up by the engineering department or the customer. Quite often these hardness specifications are made without a proper knowledge of what they mean in the heat treat and in service. Several years ago, the customary check for hardness of a case hardened part was either the scleroscope or the file. These means have been in many cases superseded by the Rockwell, and in some cases both the Rockwell and the scleroscope specifications are given as optional requirements. Many cases arise where a file test is specified, but a Rockwell or scleroscope test is substituted because of the difficulty of obtaining a satisfactory inspection by means of a file. While charts have been published comparing the readings of Brinell, Rockwell, and scleroscope, the results often do not coincide with the com-

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parative readings, especially in case hardened material. The file test, which is quite common, often does not bear a very close relation to the scleroscope or Rockwell readings, and where the specifications are rigid it is very difficult for the heat treater to successfully meet them, especially with the higher alloy case hardened steel.

It is true that there are many parts which can be checked satisfactorily with any one of the above mentioned methods, so that there is little difficulty in satisfying the inspection department. This is true of such parts as pins and pieces of uniform section made from the lower alloy case hardened steels. We have found, on the other hand, that it is practically impossible to reconcile the Rockwell, scleroscope, and file tests on some parts, and the investigation as to the reason for the difference has developed several points which seem to have practical value. It is believed that a hardness specification in any important part should be set by the metallurgical department and should be chosen to insure the maximum performance in service. Thus it will be found that certain structural conditions are much more important in their relation to abrasive wear than others, and that the same is true for maximum toughness, etc., and that the structural conditions have an effect on the choice of the hardness test which should not be overlooked.

One of the specific problems which developed a discrepancy between the various methods of checking was the difficulty in explaining the variation in scleroscope hardness on water-quenched worms made from carburized S.A.E. 3115 steel. Another case was found in case hardened S.A.E. 4615 drive pinions quenched from the carburizing pot in oil, which is the usual procedure followed in heat treating this part. Here difficulty was often met with in reconciling the file test with the scleroscope or Rockwell. Another case was found where the same steel used for case hardened axle shafts quenched from the carburizing pot was absolutely file hard with a maximum scleroscope of 65. Several other cases can be cited where the lack of agreement made it difficult to meet the hardness specifications when the scleroscope or the Rockwell or file was used as a hardness specification.

In investigating the properties of quenched S.A.E. 3120 steel used for worms, two steels were selected of the following analysis:

Steel No.	C	Mn	P	S	Cr	Ni	Si	Grain Size
2C	.25	.55	.018	.024	.53	1.28	.24	1- 3
4B	.15	.51	.018	.023	.68	1.23	.26	9-10

These steels were chosen because they represent the maximum and minimum carbon content in this specification, as well as the coarsest and finest grain size which a check of a large number of heats revealed. The samples used in these tests were all two inches in diameter by three inches long, carburized as noted below. This size was chosen because it campared with sizes used in actual practice and because it insured a satisfactory scleroscope reading.

In making this investigation, a complete set of tensile tests, impact, transverse tests, etc., were made, but these results will not be included in this report in which we are dealing entirely with the case structure and its relation to the hardness test results. Several steps were taken in the investigation as follows:

1. A comparative hardness check was made between fine grained carbides and carbides in heavy envelopes around coarse grains. This was done by quenching and checking the fine and coarse-grained steels after slow cooling in the carburizing pot to a temperature below the A_{cm} line.

2. A comparison of the above was made with carbides in solution, that is with samples quenched from the pot or reheated above the A_{cm} line.

3. A comparison was made between coarse-grained steel quenched below the A_{cm} line to develop a heavy carbide network and the same steel given a spheroidizing treatment before quenching.

4. A check was also made of samples spheroidized before quenching and reheated above the A_{cm} line to check the possible effect of the lowering of carbon content from decarburization or diffusion during the spheroidizing treatment.

5. An investigation was made of the relation between the structure and the hardness readings by comparing microscopic studies with hardness readings.

6. Both fine and coarse-grained steels carburized to give a case of eutectoid composition were reheated to the same temperature as that used for the higher carbon samples, below the A_{cm} line.

7. Both fine and coarse-grained steels were carburized to give a case of eutectoid composition and reheated above the A_{cm} line of case No. 2 and quenched.

Table I shows that the carbide network in the hypereutectoid zone, whether coarse or fine, has little or no effect on the hardness reading. Table I also shows that the fact that the carbides in the

Table I
Effect of Condition of Excess Carbides on Hardness Test

Heat	Treatment	Surface Hardness Rockwell	Surface Hardness Scleroscope	File	Rockwell .015" deep	Remarks
2C	Carb. 1700° F. Pot cooled. Reheat 1400° F. Water quench.	66-68	97-102	Hard	66	Coarse carbide network.
4B		66-68	97-102	Hard	66	Fine carbide network.
2C	Carb. 1700° F. Pot cooled. Spheroidized, 1350° F. Reheated 1400° F. and quenched.	65-67	97-102	Hard	65	Finely spheroidized carbides.
2C	Carb. 1700° F. Quenched from pot in water. Reheat 1400° F. water quench.	67-68	98-103	Hard	65	
4B		67-68	99-104	Hard	65	

finely spheroidized form have little or no effect on the hardness test. The spheroidized carbides were obtained by holding for 17 hours at a temperature of 1350 degrees Fahr. There was some tendency on samples so treated to show a very slight decarburized surface from the furnace exposure. The results obtained, as given in Table I, indicate that the hardness readings obtained by single quenching from a low temperature of a hypereutectoid network are not affected by the condition of the precipitated excess carbide. Table I also shows that a double treatment in which the carbides are put in solution by quenching above the A_{cm} point, and then precipitated in an extremely finely divided condition by reheating below the A_{cm} point, does not affect appreciably the hardness readings.

Table II shows the effect on the hardness reading of quenching at a high enough temperature so as to retain the carbides in solution. This was done both by quenching directly from the pot, and also by cooling in the carburizing pot and reheating to the carburizing temperature and water quenching. It is evident that the reheated samples are somewhat harder both to the surface Rockwell

Table II
Effect of Carbide Solution on Results

Heat	Treatment	Surface Hardness Rockwell	Surface Hardness Scleroscope	File	Rockwell .015" deep	Remarks
2C	Carb. 1700° F. Quenched from pot—water.	57-59	75-85	Hard	65	No excess evident.
4B		56-61	75-85	Hard	64	No excess evident.
2C	Carb. 1700° F. Cooled in pot. Reheat 1700° F. water quenched.	62-65	75-89	Hard	65	No excess evident.
4B		62-65	82-90	Hard	65	No excess evident.

Table III
Effect of Carbon Content on Hardness Readings

Heat	Treatment	Surface Hardness Rockwell	Surface Hardness Scleroscope	File	Rockwell .015" deep	Remarks
2C	Carb. 1600° F. Bone—24 hrs.	65-67	95-102	Touch with	62	Hyper zone not evident.
4B	Pot cooled. Reheat 1400° F. Water.	60-67	94-100	file	62	
2C	Carb. 1600° F. Bone—24 hrs.	61-65	92-99	Touch with	62	Hyper zone not evident.
4B	Quenched from pot—water.	60-65	90-100	file	62	

and the scleroscope, but are the same hardness at a depth of 0.015 inch. This condition, as will be pointed out later, is due to somewhat less retained surface austenite in the reheated samples, due to lower carbon surface caused by furnace oxidation. If the results of Table II are compared with the results in Table I, it will be noted that the surface hardness is considerably less although the hardness at 0.015 inch deep is practically the same. These results indicate that austenite is formed at the high quenching temperature when the carbides are in solution, with a result that less surface hardness is evident as the quenching temperatures and speed of quenching becomes greater.

Table III shows the effect of carbon content on the hardness readings. Here a case with no hypereutectoid zone was obtained by carburizing in a bone compound for 24 hours at a relatively low temperature (1600 degrees Fahr.) A comparison of Table III with Tables I and II shows that the lower carbon case gives a lower hardness reading than the higher carbon case when reheated and quenched at a temperature below the A_{cm} line, but gave a higher hardness reading when quenched above the A_{cm} line for steels treated as in Table I, indicating that the excess carbides, when precipitated, tend to increase the hardness readings, while quenching from a higher temperature to retain the carbides in solution will form less austenite in the lower carbon case than in a higher carbon case.

It is evident that in steels where the alloy content is sufficiently high the variation in carbon content and quenching temperature have an important effect on the case hardness reading, and also a combination of high quenching temperature and carbon content would tend to lead to an increasing amount of retained austenite which would affect greatly the relative values of Rockwell, scleroscope, and file.

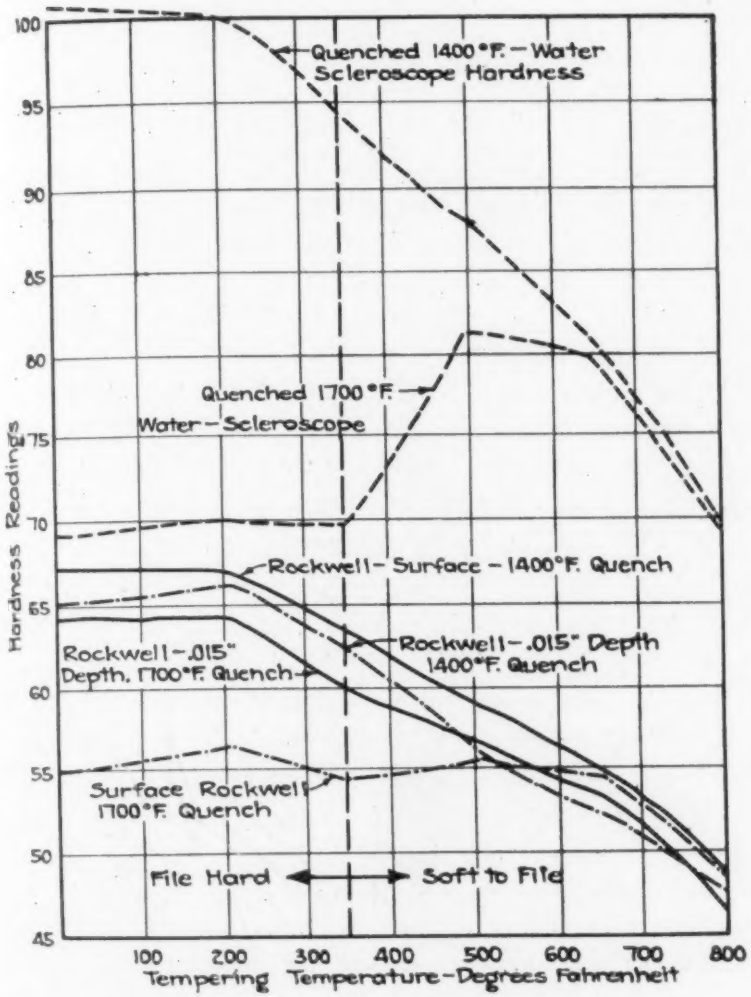


Fig. 1—Curves Showing Effect of Tempering Temperature on Hardness Readings of Austenite and Martensite.

In each of the above investigations, microscopic sections were made and the structure compared with the hardness tests. It was noted that specimens quenched from 1700 degrees Fahr., especially those containing the higher carbon showed an austenitic structure in the quenched hypereutectoid zone. In order to determine the effect of tempering and its effect on the structure itself, and on the hardness readings, samples quenched from the carburizing pot at 1700 degrees Fahr., in water were examined. The results obtained are shown in Fig. 1. In the same Fig. are plotted the results obtained by tempering the samples quenched from the pot at 1700 degrees Fahr., and reheated to 1400 degrees Fahr., for a water quench. Figs. 2,

3, 4, and 5 are microsections showing the structure obtained on the pot-quenched samples in the hypereutectoid zone at different tempering temperatures.

Fig. 2 shows the hypereutectoid zone structure as pot-quenched. Here considerable austenite is evident and the surface Rockwell and scleroscope readings are relatively low. The Rockwell reading taken 0.015 inch below the surface was 65, which is considerably harder than at the surface.

Fig. 3 shows the structure obtained after a 650 degrees Fahr. temper on the pot-quenched samples and shows a considerable increase in scleroscope reading, and a slight drop in the Rockwell reading at the surface. The Rockwell taken 0.015 inch below the surface coincides with the Rockwell of the surface, indicating that as far as the Rockwell is concerned a more uniform condition of the case has been reached. The microsection shows practically the entire elimination of austenite consisting mostly of martensite with troostite. This structure is quite soft to the file as compared to the structure in Fig. 1, which is file hard.

Fig. 4 shows the structure of the double treated 1400 degrees Fahr., quenched hypereutectoid zone and shows an extremely fine martensitic structure with high hardness readings.

Fig. 5 shows the structure of the same sample after a tempering treatment at 650 degrees Fahr., and shows a finely divided martensitic troostitic structure, almost irresolvable. It will be noted that the Rockwell hardness reading of the low temperature quench was considerably higher than the surface Rockwell obtained from the high temperature quenching, and corresponds closely to the Rockwell obtained at 0.015 inch under the surface on the pot-quenched samples. It is interesting to note that as the tempering temperature increases, the values obtained from both methods of quenching tend to coincide, indicating that the difference between the two samples is most undoubtedly due to the austenitic surface of the samples quenched from the carburizing temperature. It is also interesting to note that all samples are file hard at the 350 degrees Fahr. tempering temperature, while those tempered from a higher temperature than this become increasingly soft to the file, indicating that the high carbon austenitic condition is normally file hard, although it is undoubtedly much more ductile than the martensite obtained from the low temperature quench, which is equally file hard. There seems to



Fig. 2
Fig. 3
Fig. 4
to 1400 D
Fig. 5
Etched in

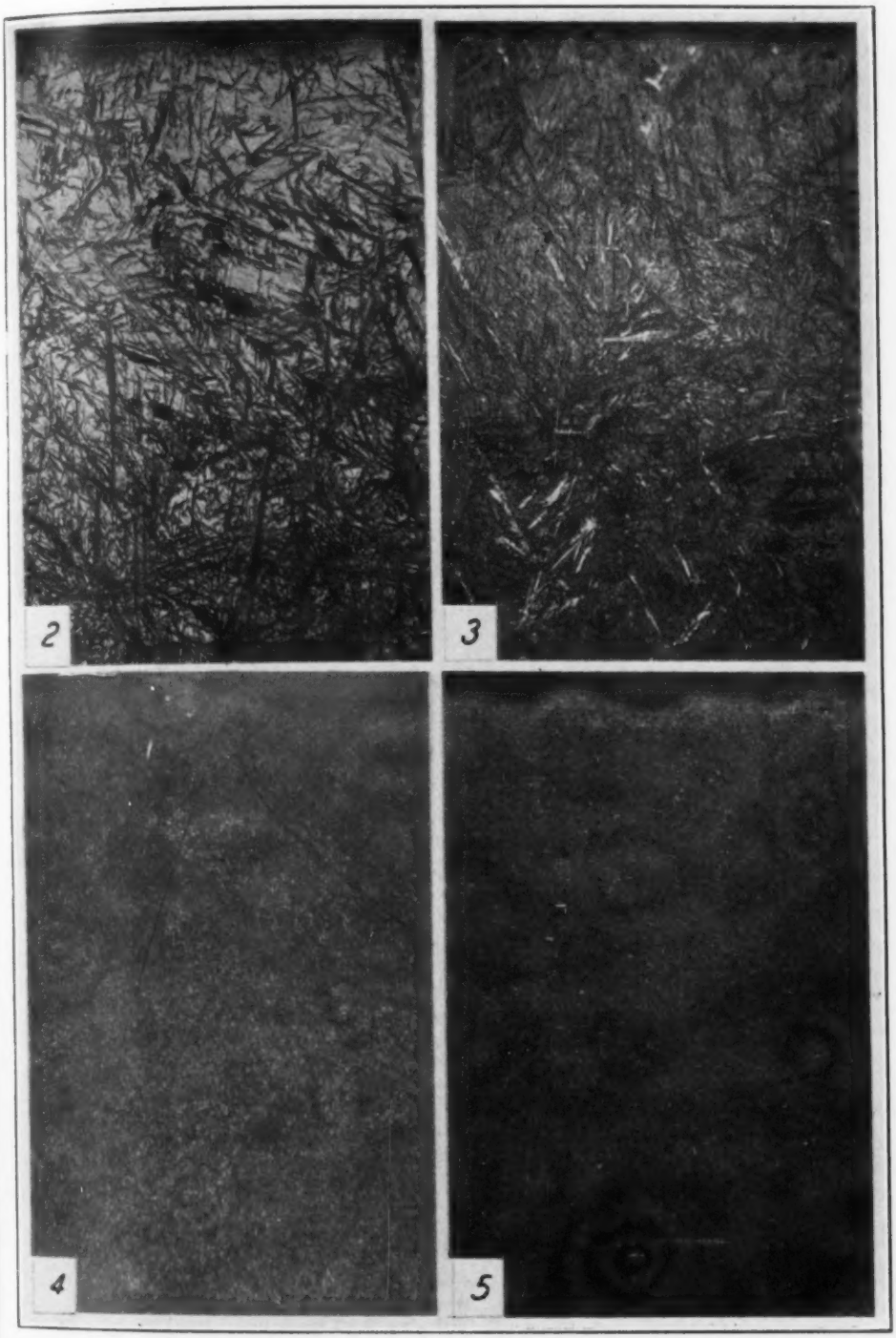


Fig. 2—Photomicrograph of Case Quenched into Water from 1700 Degrees Fahr.
Fig. 3—Same as Fig. 2 but Tempered at 650 Degrees Fahr.
Fig. 4—Section of Case Quenched into Water from 1700 Degrees Fahr., Reheated to 1400 Degrees Fahr., Quenched in Water.
Fig. 5—Same as Fig. 4 After Tempering at 650 Degrees Fahr. All Specimens Etched in Nital. × 100.

Table IV
Effect of High Temperature Quench on Hardness Readings of High Carbon Alloy Case

Heat	Treatment	Surface Hardness Rockwell	Surface Hardness Scleroscope	File	Rockwell .015" deep	Remarks
2C	Carb. 18 hrs., 1700° F. 4 hrs.,	28-32	32-42	Hard	61	Case quite ductile.
4B	2000°F. Quench from pot—water.	32	40	Hard	61	

be little evidence that the austenite can be changed to martensite by tempering and the contrary is apparently true indicating that the austenite changes directly to troostite or that the change from austenite to troostite through martensite is extremely rapid.

From Fig. 1 it is also evident that it is possible under the right conditions, to obtain a file hard surface with any scleroscope reading above 70, depending upon the structural condition of the case after quenching, and also that it is very easily possible to increase the scleroscope hardness by tempering, but that this is not at the same time possible with the Rockwell reading.

In order to obtain a still greater concentration of austenite, samples of both fine and coarse heats were carburized for 18 hours at 1700 degrees Fahr., and then raised to 2000 degrees Fahr., for 4 hours and quenched in water from this temperature.

Table IV shows the results obtained, which are quite interesting in that they show that it is possible to have a file hard surface with a Rockwell on the surface as low as 28, and a scleroscope reading as low as 32. In the same samples, readings taken 0.015 inch below the surface showed a hardness on the Rockwell of 61, indicating the presence of considerably more martensite than at the surface.

It is interesting to note that samples from this lot which were exposed by the shrinkage of the carburizing compound, and therefore decarburized on one side, showed a variation in scleroscope reading from an average of 40 on one side to an average of 90 on the other (decarburized) side. The corresponding Rockwells are 32 for the undecarburized surface, and 60 for the exposed surface. At the same time it was found that the low Rockwell and scleroscope reading corresponded to a decidedly file hard surface, whereas the high scleroscope and Rockwell readings corresponded to a decidedly soft surface to the file. These conditions were due to the presence of austenite in the high temperature quenched high carbon surface.

and the absence of austenite in the low carbon exposed surface quenched from the high temperature. This indicates the importance of the carbon content as far as the austenite is concerned in this particular analysis.

Conclusion

Hardness specifications are generally designed to produce on the part being hardened, a surface which will in service give the longest life in the most economical manner. The great variation of surface requirements met with in the automotive industry make it difficult to specify a certain hardness by any particular measuring device, and be sure that the hardened surface will be the most satisfactory for the use intended. Thus the surface which must be obtained on highly stressed gears, bearings, etc., where resistance to abrasion and exceedingly high unit pressures are met with, should be different than those required on a worm or piston pin surface where the unit pressures are relatively low and the most important requirement is the resistance to wear caused by sliding friction. Another case where a special surface hardness is required is met with in crusher jaws, steam shovel jaws, etc., where the surface is subjected to extremely heavy abrasion and considerable battering and pounding. Thus it has been found for gears, bearings, etc., that some sacrifice in absolute hardness as measured by the Rockwell or scleroscope, must be made to obtain the necessary ductility to insure resistance to extremely high alternate stresses. On surfaces such as the automotive worm, the surface should be as hard as possible to resist wear and it is not as a rule necessary to give special concern to obtaining ductility at the sacrifice of surface hardness. On parts which must resist considerable battering it has been found that the austenitic structure such as is obtained with the Hadfield analysis, is almost impossible to improve on. It is quite possible that many cases where an extremely high scleroscope or Rockwell reading is specified, that the softer (to these instruments) austenitic surface, which is file hard, would give much better results in service. At least this is indicated by the results obtained with pot-quenched case hardened alloy drive pinions.

An investigation is now being made to determine as accurately as possible the relative values of the austenite and the martensite surface in resisting wear and highly stressed alternate

or intermittent loads. It is believed that the maximum amount of excess carbides in a case together with the maximum Rockwell and scleroscope hardness, combine to give the maximum resistance to pure abrasive wear so that this condition is most satisfactory for worm and similar surfaces. It has been found that the nitrided surface is superior to the case hardened surface for abrasive service, but it has not been found that this is true (as yet) for gears and similar applications. It is recommended that the selection of the hardness ranges for any particular application be considered much more carefully than is usually done, particularly with the higher alloy case hardened steel and that where two ranges are given or where the file is specified, that the conditions of quench, tempering, carburizing practice, etc., be well known since each factor plays an important part in the character of the case obtained.

DISCUSSION

Written Discussion: By E. G. Mahin, professor of metallurgy, University of Notre Dame, Notre Dame, Ind.

The authors have called attention to a most annoying difficulty encountered in physical testing—the correlation and interpretation of hardness tests as made by different methods. Experimental tables and formulas, both empirical and theoretical, have been proposed for calculating hardness numbers from one to another of those obtained by use of different testers, but the common experience of other metallurgists is as the authors state—that these tables and formulas do not work out well in varied practice.

When we come to the matter of case hardened steels, the difficulty in reconciling tests becomes still more apparent. Indeed, it is my opinion that no hardness test that has yet been devised can possibly give more than empirical results when applied to case hardened articles, for the simple reason that all involve deformation of the surface in some way and then the hardness of supporting layers comes into consideration. Since hardness decreases more or less continuously from surface to the inner boundary of the case, the observed hardness number cannot be expected to express the actual hardness of the extreme surface, but only the *effective* hardness, *under the conditions of the method employed for testing.*

Assuming that a truly scientific testing machine is used (if such an one has ever been constructed) and that other factors are under proper control, it would appear that the method of test which involves least actual penetration of the steel piece would approach most nearly to theoretically correct values. However, this leads to the use of very small penetrators, acting under light total loads, and this brings in another variable, due to the fact that the penetrator may rest upon grains of different degrees of hardness in successive tests. For this reason the Brinell numbers should most nearly express average hardness, the Rockwell "B" numbers next, and such numbers as

those obtained by the Rockwell "B scale", the scleroscope or the Vickers testers should be most variable, when applied to steel uniform in composition and hardness from surface to center. For case hardened articles, I feel that the Brinell test is practically ruled out, unless the case is very deep, for reasons already stated.

The file test belongs in that peculiar class of testing methods so often found in industry—an empirical test that is practically valueless in the hands of an amateur but capable of giving valuable information when used by one who has had much experience. Variables which cannot be eliminated are the quality of steel in the file, the degree to which the file has been worn (after the very first stroke, the file is altered in this respect), the speed of travel and the pressure applied. If the file, by excessive pressure, can be made to bite through the hardest exterior layer, it will cut the softer layers underneath. This is particularly true if the test is applied to a curved surface or to a corner.

The one who can devise a really scientific method for determining the surface hardness of case hardened material will deserve the gratitude of all metallurgists. The authors of this paper have done well in calling attention to the fact that it is *effective* hardness, *manifested under the service conditions in use*, that is important, rather than actual hardness, which continuously decreases as the exterior layer is passed. It is especially for this reason that plant practice may employ any one of several methods with fairly satisfactory results, provided that specifications have been evolved for given parts, based upon long experience with service requirements.

Written Discussion: By A. L. Boegehold, chief metallurgist, General Motors Corp., Detroit.

We were particularly pleased upon reading the paper by Messrs. McQuaid and McMullan dealing with variation of case hardness after different heat treatments because the subject is along lines similar to some work which has been conducted in our laboratories with similar results. We wish to supplement Messrs. McQuaid and McMullan's paper by presenting some results showing how surface hardness after quenching is dependent upon how composition as well as treatment influences the amount of retained austenite.

Fig. 1 shows the hardness penetration curve for S.A.E. 1020 steel carburized and water-quenched from 1400 degrees Fahr. after heating at that temperature in Aerocase.

The increase in hardness in the first 0.010 inch under the surface of the S.A.E. 1020 steel water-quenched from 1400 degrees Fahr. differs from the authors' results showing hardness at the surface greater that at a depth of 0.015 inch in Table I, page 4.

Fig. 2 shows a curve for a 2.75 per cent manganese, 0.22 per cent molybdenum steel carburized and oil-quenched from 1550 degrees Fahr. heated in Aerocase.

The reason for the low hardness at the surface of the manganese-molybdenum steel can be seen in Fig. 3 which shows the case microstructure of this steel magnified 750 times. There are large patches of austenite to a depth of 0.010 inch below the surface. Upon tempering this sample the surface hardness increased until 900 degrees Fahr. was reached. Fig. 4

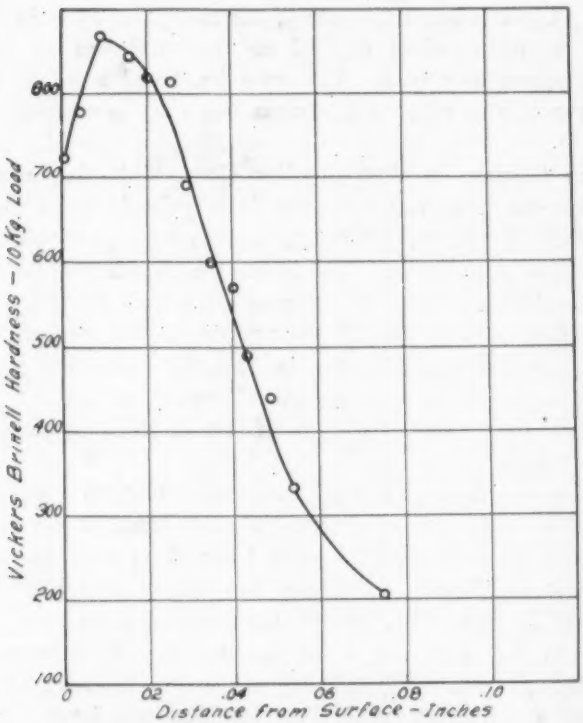


Fig. 1—Hardness Penetration Curve for a Carburized S.A.E. 1020 Steel, Water Quenched from 1400 Degrees Fahr.

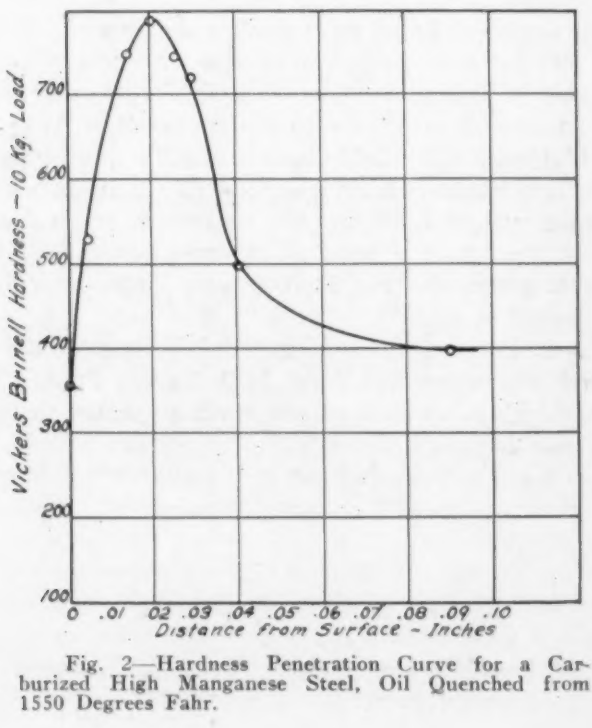


Fig. 2—Hardness Penetration Curve for a Carburized High Manganese Steel, Oil Quenched from 1550 Degrees Fahr.

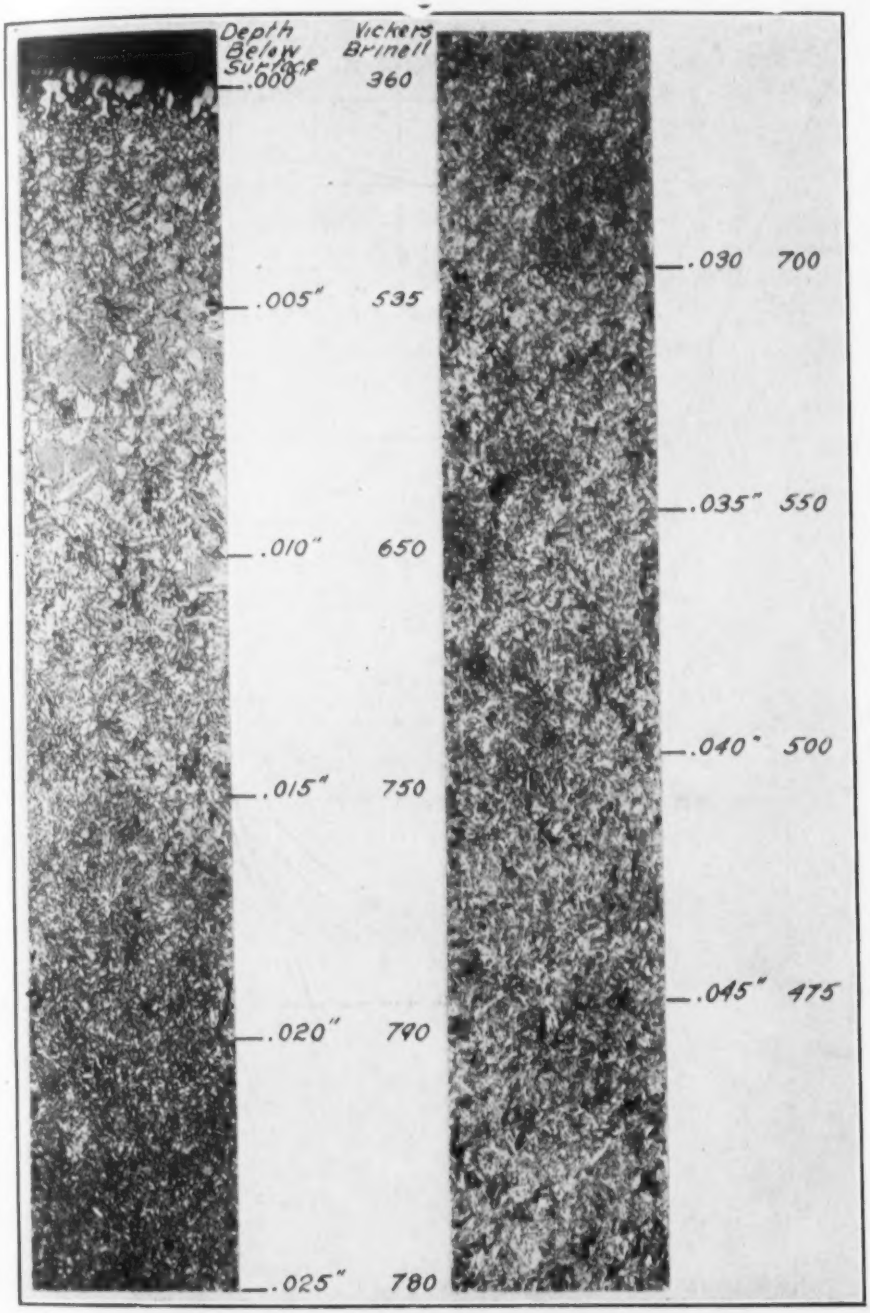


Fig. 3—Microstructure of Case Hardened Manganese-Molybdenum Steel. Original Magnification × 750.

shows the effect of tempering temperature on the hardness. At a hardness of 645 Vickers Brinell after a 900 degrees Fahr. temper, the sample could be readily filed, while in the quenched condition with a hardness of 360 Vickers

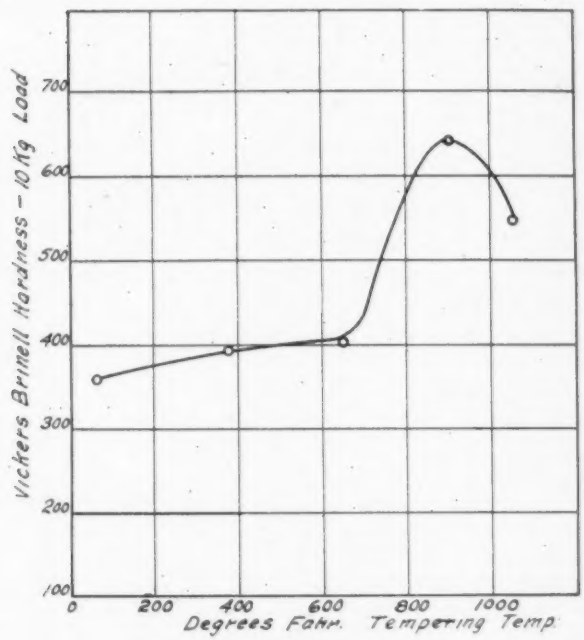


Fig. 4—Hardness Penetration Curve for a 2.75 Per Cent Manganese-Molybdenum Steel Carburized and Oil Quenched from 1550 Degrees Fahr. Tempered at Various Temperatures.

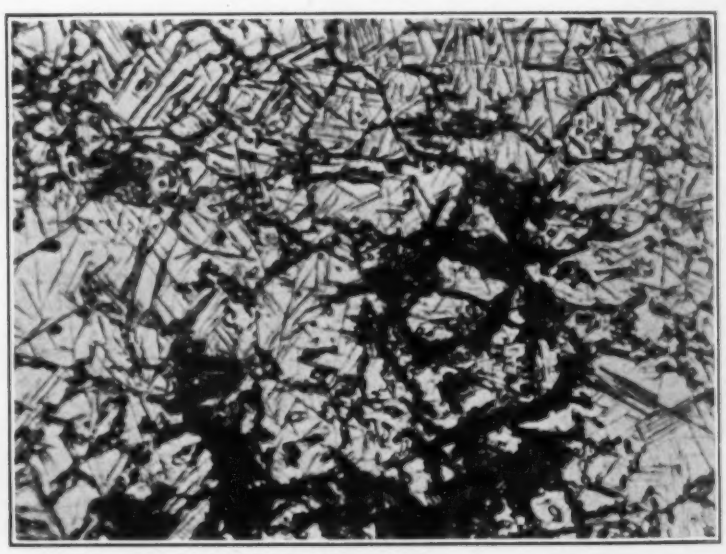


Fig. 5—Manganese-Molybdenum Steel Case Hardened and Tempered at 850 Degrees Fahr. × 750.

Brinell at the surface it was possible to barely mark the surface with a file, which confirms the authors' findings that file hardness cannot be reliably determined with indentation hardness testing machines.

The statement that austenite does not change to martensite on tempering or that it passes through this stage with great rapidity is not true of the high manganese-molybdenum steel in question which showed that a distinct martensitic structure had taken the place of austenite after an 850 degrees Fahr. temper. See Fig. 5.

Written Discussion: By J. A. Comstock, assistant metallurgist, Peoples Gas Light and Coke Co., Chicago.

The authors are to be complimented on their very interesting and logical presentation of the facts pertaining to the heat treatment of carburized alloy steels to obtain suitable hardness for wear resistance. The writer's own experience in several instances absolutely coincides with the authors' reported findings, and it is gratifying to read so logical an analysis of the problem.

In dealing with alloy carburizing steels with as much as 3-5 per cent nickel content, we have observed that for rates of cooling from the carburizing temperature equivalent to normalizing treatment, an austenitic surface layer is obtained, which, were it not for its hardness of somewhat more than ferrite, could be easily mistaken for a decarburized surface on etching. Reheating to a temperature considerably lower than the carburizing temperature but above the Ar critical point and quenching, produces a martensitic surface.

Regarding the effect of precipitated carbide particles, we have for some time been of the belief that a structure comprising finely dispersed carbide particles in a martensitic or martensitic-austenite matrix exhibits the theoretical features of a bearing metal in its physical make-up having very hard particles supported in a hard and tough matrix. Such a structure would be desirable for parts like bearing races provided that the amount and distribution of the precipitated particles is controlled within certain limits to obviate the trouble of spalling of the surface when the matrix is too highly contaminated with these carbide particles.

We hope the authors will see fit to publish the results of their investigation regarding the type of structures most desirable for various wear-resistant parts.

Authors' Closure

We appreciate very much the discussions submitted, and are especially pleased to find that they contain little, if any, adverse criticism.

I believe that the reason Mr. Boegehold shows a falling off in hardness at the surface as measured by the Vickers on the sample of carburized S.A.E. 1020 steel after a water quenching at 1400 degrees Fahr., may be due to the more sensitive hardness checks which are obtained with the Vickers tester. On the other hand, it is also possible that the formation of austenite at this temperature in this steel may be obtained by heating in Aerocase which might possibly result in the formation of nitrides in the surface layers of the steel. This is quite possible and similar results have been obtained in heat treating case hardened alloy steels at low temperatures in a heating bath containing approximately 40 per cent sodium cyanide.

Mr. Comstock brings up the point that an ideal bearing metal would probably be one in which we had the carbides in a fine grained martensite.

That all depends on what you mean by bearing metal, whether you are talking about a roller bearing or an ordinary sliding bronze against steel. While we insist upon fine grain we prefer to have a fine grained austenitic structure for roller bearings and gears rather than a fine grained martensitic structure which is, in our opinion, somewhat less ductile. I do not want to get into a long discussion about grain size, as you know that is one of my favorite subjects. But the coarse grained steels which harden more deeply and more easily will give you a different hardness condition more suitable for sliding bearings where resistance to abrasion is important, than the finer grained steel of the same analysis under the same quenching conditions. I am sure that the finer the grain size of the steel can be and still get the required hardness the more satisfactory the results are going to be in service.

OXYGEN IN STEEL

By Marcus A. Grossmann

Foreword

Oxygen in Steel, an account of some behaviors of oxygen in solid steel, including evidence on the solubility of oxygen in steel at heat treating temperatures, evidence that such dissolved oxygen causes the steel to have a lesser tendency to harden when quenched, and evidence that "abnormal" carburizing steels owe their abnormal structures to oxygen absorbed in carburizing.

IN a memorial lecture it seems fitting and proper to choose for discussion a subject which was once of interest to the man whose memory is to be honored. In the case of Edward De Mille Campbell, this offers a very wide range of topics, for there were indeed an extraordinary number of subjects in which he not only was interested, but could speak with authority. In the present case, we have elected to discuss oxygen in steel, and while it is not surprising to find even this subject included in Prof. Campbell's activities, it is nothing less than amazing that he should have expressed opinions that are even today unimpeachable, in a field of endeavor which during his lifetime was scarcely begun. We shall have occasion to quote Prof. Campbell later.

We have, as stated, elected to discuss oxygen in steel, and in particular some of the ways in which oxygen, when present in steel, affects certain properties of that steel. We shall be concerned only with the oxygen found in solid steel, and not its behavior in the liquid metal.

Now there are numerous forms which oxygen may take when it occurs in solid steel, and furthermore the total amount present may vary within wide limits. The tasks that confront us in any particular lot of steel are, then, first to determine accurately the total amount of oxygen present, then to discover the various forms among which that total oxygen is distributed in that particular lot, and finally to state how each form affects the behavior of the steel. It is our

This is the fifth Edward De Mille Campbell Memorial Lecture, presented by Dr. Marcus A. Grossmann, vice-president, Republic Research Corporation, Massillon, Ohio. Lecture presented at the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930.

purpose today to review briefly the work that has been done up to the present time, and then to describe certain experiments and offer certain new data which have seemed to have considerable bearing on the whole general problem.

If we discuss these three aspects of the subject in order, we have first to mention the various analytical methods employed for determining the amount of oxygen present in steel. We shall find that there is up to the present time but one method which is wholly reliable for determining the *total* oxygen, namely the method developed by Jordan which involves melting in a carbon crucible in vacuum, but we shall see also that there are numerous other ingenious methods, many of them useful for determining a particular form of oxide in the steel.

The earliest method was the well-known one devised by Ledebur[1] in about 1880, in which the steel is heated in a stream of hydrogen, the reaction of the hydrogen in the stream with the oxygen in the steel forming water which is collected and weighed. The method is useful for determining dissolved oxygen and the easily reducible oxides like iron oxide, and so will determine practically all of the oxygen in some certain steels.

In a modification of this method, the steel sample dissolves in a molten alloy of antimony and tin, while at the same time applying the stream of hydrogen at high temperature in the usual manner. Dr. Desch of Sheffield, England, and Mr. Jordan of Washington have kindly permitted us to report their results here, Dr. Desch having written recently that on a sample of open-hearth iron which by the vacuum fusion method in Washington and in Aachen showed 0.058 and 0.059, Dr. Desch found 0.055 by this method.

These two methods, the latest and the earliest, the vacuum fusion method and the hydrogen method, seek to extract the oxygen from the steel, in converting it to a gaseous compound, and subsequently to absorb and weigh this gaseous compound. The other methods seek to isolate the oxides which actually occur in the steel, by dissolving away the metallic iron and leaving as residue these oxides alone. These residue methods include a number of instances of ingenious technique and are at times most useful, so that they call for some mention.

It seems appropriate to record at this point the fact that many of

[1]Figures refer to bibliography appended to this lecture.

the analytical methods for oxygen have been subjected to critical study in the past few years at the University of Aachen. The late Prof. Oberhoffer instituted there an extensive program of investigation, which has now come to fruition and which has yielded invaluable information on methods of oxygen analysis.

An early method of residue analysis involved treatment of the steel sample with iodine solution. The iodine was intended to dissolve away the metallic iron, and since there was but little reaction of the iodine with the oxides, these oxides were supposed to be left as residue. It was found later by Oberhoffer[2] and his associates that iodine can to advantage be replaced by bromine, with improvement in accuracy.

Another residue method involves heating the steel sample in a stream of chlorine gas, as reported especially by Wasmuht.[3] If the heating is done at a suitably low temperature, say 400 degree Fahr. (200 degrees Cent.), the metallic iron forms with the chlorine a gaseous compound, a volatile chloride which passes off as gas, leaving behind as residue the oxides which were in the steel, or leaving at least substantially all such oxides.

Still another residue method consists in dissolving the steel sample in cold dilute nitric acid. This method, known as the Dickenson[4] method, and studied also by Herty, has been varied by using sulphuric acid, and in either case will leave as residue particularly the silicates and aluminates.

In all of the residue methods, it is the aim to use a sufficiently large sample so that the residue, which one fondly hopes comprises the original oxide inclusions, can be not only weighed, but also analyzed for its constituent elements. The residue methods have yielded much extremely useful information, especially because they make it possible to collect and examine the actual inclusions which were in the steel. There are certain discrepancies among the methods, since an analysis of a steel by one residue method will show a certain quantity of inclusions of a certain composition, whereas another residue method may indicate a different amount of inclusions of a somewhat different composition, in the same steel. But these discrepancies have been studied extensively, and are believed to be due to the fact that the reagent which dissolves away the iron introduces two sources of error. The reagent has a slight dissolving action on the inclusions, and this dissolving of the inclusions varies

with the different reagents. But furthermore, and perhaps more important, the reagent seems in certain cases to oxidize some of the metal, which may then in its oxidized form be found to accompany the actual inclusions as oxide, instead of being dissolved away as metal. The apparent amount of inclusions is thus higher than the actual amount present.

Considering all these circumstances, then, a reliable method for determining oxygen is of paramount importance.

The method on which reliance is now placed is the vacuum fusion method already mentioned, which was developed by Jordan[5] at the Bureau of Standards, and which has since been studied extensively, especially by Hessenbruch[6, 7] and by Diergarten[8, 9] both in Aachen.

This vacuum fusion method, although it does not distinguish between among the various kinds of oxides present, does determine the total amount of oxygen. The sample of steel is heated in a graphite crucible to its melting temperature and beyond. At this high temperature the oxides in the now molten steel react with the carbon derived from the graphite crucible, forming carbon monoxide, CO gas. The carbon monoxide gas passes off due to the vacuum, is oxidized to carbon dioxide, CO_2 gas, and is then absorbed and weighed. The method has but few limitations, and can on the whole be relied upon for accurate work.

* * *

A satisfactory method of analysis being thus available, we are in a position to determine what content of oxygen is usual in steel. A general idea of how much oxygen may be expected, will serve as a guide in any particular case, indicating whether the analytical result obtained (the oxygen content) is normal, or is unexpectedly high or low. If, however, we make for example several hundred determinations, we find that there is no general normal which applies to all steels, but that on the contrary there are different values to be expected, depending both on the composition of the steel and on the manner in which it was made.

A number of representative values are given in an accompanying table, Table I, from which a few typical figures have been selected for the slide (Fig. 1).

It will be seen that a representative value for low-oxygen steels would be around 0.006 to 0.008 per cent, and that steels of the high-oxygen type would contain perhaps 0.040 to 0.060 per cent. Other

Table I
Typical Oxygen Contents of Commercial Steels

Number	C	Mn	Si	Cr	Ni	Cu	Mo	V	Oxygen
6545	0.44	0.57	0.17	1.07			0.33		0.0036
60154	0.31	0.64	0.17	0.85	0.16			0.19	0.0042
8496	0.30	0.77	0.17	0.90			0.14		0.0054
51572	1.08	0.38	0.28	1.50	0.18				0.0051
6836	0.18	0.59	0.18	0.59	1.17				0.0054
7406	0.39	0.66	0.18	1.07			0.20		0.0057
15109	0.54	0.73	1.98						0.0069
2151	0.28	0.51	0.18	0.98			0.34		0.0072
C.S.	0.19	0.37	0.11						0.008
A.S.	0.40	0.65	0.23	0.60	1.25				0.008
62147	0.19	0.39	0.11						0.0083
32093	1.00	0.36	0.28	1.50	0.21				0.0097
92528	0.13	0.42	0.13						0.0121
S.S.	0.04	0.28	0.008			0.26	0.12	{Rim {Core	0.022 0.033
2102	0.09	0.40	0.005						0.0385
2036	0.10	0.40	0.003						0.0562
S.S.	0.03	0.18	0.01					{Rim {Core	0.019 0.068
70632	0.07	0.37	0.001						0.070
O.H.	0.02	0.03	0.005					{Rim {Core	0.047 0.103

	C	Mn	Si	Alloys	Oxygen
Carbon steel	.19	.37	.11		.008
Alloy steel	.40	.65	.23	Ni 1.25 Cr .60	.008
Soft steel	.04	.28	.008	Cu .26 Mo .12	Rim .022 Core .033
Soft steel	.03	.18	.01		Rim .019 Core .068
Open hearth iron	.02	.03	.005		Rim .047 Core .103

Fig. 1—A Few Typical Oxygen Contents in Steel, Selected from Table I.

types have intermediate values, say from 0.010 to 0.030 per cent. It may be pointed out once more that these are the normal values for the several classes of steel, and when we speak of classes of steel we mean both the composition and the manner of solidification, whether high alloy or low alloy, and whether effervescing or killed steel. Steels are found to deviate as much as perhaps 30 per cent from the normal for their class, but no instance has been observed where a type of steel containing normally say 0.040 per cent contained as

little as 0.008, or a steel containing normally 0.008 per cent contained as much as 0.040. For comparison with other values to be cited presently, it might be well to keep these figures in mind, namely the range from 0.006 to 0.060 per cent.

These comments on oxygen variation refer to large lots made on a commercial scale. Small heats made on a laboratory scale may vary much more widely, because the rapid solidification of a small heat prevents the separation of inclusions, and so traps oxides which might otherwise have time to be eliminated. The figures given above refer only to steels made on a commercial scale, cast into ingots weighing from 1½ to 5 tons each.

* * *

We have mentioned thus briefly the amounts of oxygen commonly encountered in steels of various types. But a mere stating of the oxygen content of a piece of steel has no significance unless we know the effect of the oxygen on the steel. This is the question which we propose mainly to discuss, namely the effect of contained oxygen on the properties of steel.

One phase of this question has in recent years been the subject of considerable study. A mention of oxygen in steel is likely to bring to mind first of all the oxides, the nonmetallic inclusions, the particles which can be observed under the microscope. Of course pieces of steel are sometimes found which contain still larger inclusions, like the so-called slag particles which are found in pipe cavities. But in general a reference to oxides is taken to mean the particles which are observable under the microscope and which generally arouse at least a shudder in the observer.

Indeed the common assumption, that the presence of such oxide particles might affect the mechanical properties of the steel, seems reasonable enough. As a result a number of investigators have in recent years worked on this problem, attempting to establish what size of particle is definitely harmful, and what the limiting size is, below which other effects are likely to overshadow any effect of the oxide. It will be realized that such a study would call for an extremely careful examination of mechanical properties. Investigators have carried out an enormous amount of work on the effect of inclusions, both as to the numerical value of the mechanical property studied, and as to the manner of failure of pieces tested to destruction.

But there is also another direction in which we may look for effects of oxygen on the properties of steel. Steel is an alloy of iron

and carbon, and the properties of steel are due in large measure to the behavior of the carbon and its effect on the iron. It is here that we may look for an effect of oxygen, namely its possible influence on the behavior of carbon in steel-treating reactions. And so your speaker has led you triumphantly to the subject that he really wishes to talk about.

We propose to offer for your consideration evidence showing that oxygen when present in certain forms affects the behavior of carbon in steel treating reactions, that is to say, it affects the rate at which steel will harden, the rate at which it will decarburize, and the rate at which certain microstructures make their appearance. But these effects seem to be noticeable only if the oxygen is in a certain form,—other forms seemingly do not behave in the same way.

Now if one form or mode of occurrence of oxygen in steel affects carbon reactions while other forms do not, it would seem probable that if oxygen could *dissolve* in iron, this dissolved form would be the effective one. Since this seems an interesting point, let us consider a little the evidence on oxygen solubility, before proceeding to the effect of oxygen on carbon behavior and steel reactions.

In the first place it will be recalled that the original Ledebur[1] method, for determining oxygen in steel, involved heating the sample at say 1830 degrees Fahr. (1000 degrees Cent.), in a stream of hydrogen, the oxygen of the steel reacting with the hydrogen to form water vapor which was weighed. If we conceive a mechanism by which such a reaction could take place, we find most plausible a diffusion of the oxygen from the interior of the sample to the surface, at which point the oxygen atoms are free to combine with the hydrogen, and if we think of diffusion of the oxygen from the interior of the sample to the surface, we can hardly escape the conclusion that if the oxygen diffused, it had to dissolve in the iron to do so.

But if this reason for assuming oxygen solubility seems to some a bit unconvincing, as perhaps it may, there is at hand a group of data which we presumptuously hope you will consider imposing, and which points to oxygen solubility in steel because oxygen is shown to be absorbed into the steel during a heat treatment, that is to say,—the quantity of oxygen contained in the steel is increased. Here, too, and perhaps more obviously, one can hardly avoid the conclusion that oxygen dissolves in the iron and that it is for this reason that it is absorbed.

The heat treatment to which we refer, which leads to absorption of oxygen into the steel, is pack carburizing, that is, carburizing in a packing mixture of the charcoal type. If we wonder how such a carbon mixture could lead to absorption of oxygen, we must remember that the actual carburizing agent in such a mixture is prob-

Table II
Absorption of Oxygen in Pack-Carburizing

Heat No.	C	Mn	Si	Ni	Cr		Oxygen	Increase
Electrolytic iron							0.0275	
Electrolytic iron carburized							0.0344	0.0069
A	0.04	0.12	0.009			0.25 Cu	0.047	
A carburized							0.081	0.034
B	0.03	0.05	0.003			0.04 Cu		
							{0.036	
							{0.033	
B carburized								
							{0.078	
							{0.077	0.043
C	(0.02)	0.02	(0.01)			0.05 Cu	0.031	
C carburized							0.059	0.028
7037	0.17	0.58		1.19			0.0037	
7037 carburized							0.0101	0.0064
2543	0.18	0.59	0.20	3.39			0.0072	
2543 carburized							0.0201	0.0129
6836	0.18	0.59	0.18	1.17	0.59		0.0054	
6836 carburized							0.0113	0.0059
6719	0.17	0.64				0.23 Mo	0.0087	
6719 carburized							0.0222	0.0135
4736	0.13	0.48			0.74		0.0071	
4736 carburized							0.0230	0.0159
1887	0.15	0.56			1.00	0.20 V	0.0070	
1887							0.0067	
1887 carburized							0.0098	
1887 carburized							0.0116	0.0038
4228	0.12	0.40		0.49			0.0024	
4228							0.0026	
4228 carburized							0.0102	
4228 carburized							0.0086	0.0069
600	0.10	0.50					0.0075	
600 carburized							0.0167	0.0092
270	0.11	0.47	0.31			0.11 Mo	0.0047	
270 carburized							0.0105	0.0058
602	0.10	0.75				0.36 Mo	0.0042	
602 carburized							0.0117	0.0075
289	0.11	0.96	0.32			0.48 Mo	0.0302	
289 carburized							0.0370	0.0068
296	0.16	0.86	0.23			0.61 Mo	0.0124	
296 carburized							0.0135	0.0011
296A	0.16	0.99	0.29			0.84 Mo	0.0117	
296A carburized							0.0150	0.0033

ably carbon monoxide CO gas, the charcoal being the source from which this gas is generated. In the carburizing by this carbon monoxide gas, the carbon splits off from the gaseous compound, dissolves in the iron and is absorbed, leading to the familiar rise in carbon content. And the interesting point for our present consideration is that a certain amount of the oxygen seemingly splits off in the same way and is absorbed in the steel, for there is an increase in the oxygen content also.

Since the first discussion of this fact two years ago,[10] there has been much confirmatory evidence. Table II printed herewith lists a number of our own results, showing how the oxygen content of various samples increased during carburizing by amounts varying

				Oxygen		
Steel	C	Mn	Alloy	Before	After	Absorbed
A	.02	.03		.031	.052	**.021**
B	.10	.25		.026	.031	**.005**
C	.18	.59	1.17 Ni .59 Cr	.005	.011	**.006**
D	.13	.48	.74 Cr	.007	.023	**.016**

Fig. 2—Oxygen Absorption in Carburizing (a few representative values from Table II).

from .001 to .028 per cent. A few representative values from this table (Table II) are given in the slide (Fig. 2).

Of other work, the most extensive list we have found is that of Dr. Treinen[11] of Aachen, Germany, whose data are reproduced here by kind permission of Director Eilender of that University, and are given in an appended table (Table III). A few representative figures are given in the next slide (Fig. 3), and we note oxygen absorption in every case.

Table III
Oxygen Absorption in Carburizing (Treinen)

		Oxygen		
Material	Time and Temperature	Before carburizing	After	Increase
Electrolytic iron reduced in H_2 and then annealed	24 hrs at 950° C	0.0181	0.0202	0.0021
Electrolytic iron from Heraeus	24 hrs at 950° C	0.0204	0.0229	0.0025
Oxygen-bearing material	21 hrs at 950° C	0.160	0.187	0.027
Another oxygen-bearing material	21 hrs at 950° C	0.225	0.214	(decrease .011)
Various	12 hrs at 950° C	0.029	0.042	0.013
		0.042	0.055	0.013
		0.049	0.061	0.012
		0.059	0.070	0.011
		0.067	0.080	0.013
		0.092	0.098	0.006

A few further data, published by Diergarten[9] of Aachen, are given in the next slide (Fig. 4) and show the same effect.

Furthermore, we shall have occasion presently to discuss a factor which influences the *amount* of oxygen absorbed in carburizing, and

Material	Oxygen Before	Oxygen After	Oxygen Absorbed
Electrolytic iron, treated	.018	.020	**.002**
Oxygen-bearing material	.160	.187	**.027**
Various	.029	.042	**.013**
	.092	.098	**.06**

Fig. 3—Oxygen Absorption in Carburizing (from Treinen's data, Table III).

	Oxygen Before	Oxygen After	Oxygen Absorbed
Electrolytic iron	.021	.023	**.002**
	.020	.023	**.003**
Electrolytic iron,) hydrogen-treated)	.018	.021	**.003**
	.018	.020	**.002**

Fig. 4—Oxygen Absorption in Carburizing (Diergarten).

in that connection will cite a group of ten further steels, all of which showed an increase in the oxygen content.

If this listing of results has savored of "Methinks he doth protest too much", we must reply that the phenomenon was unexpected and that it leads to far-reaching results which will be discussed presently, and we therefore welcomed confirmatory evidence.

* * *

At any rate, the purpose in presenting the data was to introduce the conception of soluble oxygen. But the question whether the oxygen is truly in the dissolved state is a theoretical matter awaiting further evidence. A more practical point, to which we now come, is

the effect of oxygen on the hardening and the structure of steel. Here the facts speak for themselves, and we shall find, like the rain which falls alike on the just and on the unjust, that oxygen affects alike the steel of the technician and of the theorist, whether the person doing the steel-treating thinks *his* oxygen is dissolved or precipitated, or even colloidal.

Now the easiest way to look for an effect of oxygen on the hardening of steel is, obviously, to compare steels which are alike in composition except for their oxygen content. But the securing of steels which are alike except as to oxygen content, is difficult for the following reason: as a result of the melting processes, the steels likely to be high in oxygen are those in which only small amounts of deoxidizers like manganese and silicon have been used. If the oxygen content is to be low, important amounts of these deoxidizers must be used during the making of the steel, and as a result residual manganese and silicon are found in such steels. Hence one finds almost universally that steels low in oxygen are relatively high in manganese and silicon, whereas steels high in oxygen are generally low in these elements.

In order to secure steels substantially alike in manganese and silicon, and yet varying in oxygen content, the following simple circumstance was here made use of: it is known that in effervescing or so-called "open" steels, that is, steels which give off gas during the solidification of the outer portion of the ingot, the inner portion of the ingot is higher in oxygen content than the outer portion, that is, the core is higher in oxygen than the rim. This difference in oxygen content between core and rim is due to the different manner of solidification in the two portions of the ingot. We thus have in the same ingot two portions of the same mass of steel, the two portions presumably alike in other elements, but differing in oxygen content. A billet from an ingot of such steel was selected for test, and was analyzed, with the results given in the next slide (Fig. 5).

We see that the carbon, manganese and silicon are practically identical in the two portions, but the oxygen is three times as high in the core as in the rim. An etched cross-section of the billet is shown in the next slide (Fig. 6).

In order to compare the hardening quality or hardening power of the rim and the core, and so discover a possible effect due to difference in oxygen content, a special hardness-testing block was used. This block originated with Rawdon and Epstein at the Bureau

	C	Mn	Si	Oxygen
Rim	.02	.18	.005	**.017**
	.02	.18	.01	**.020**
Core	.032	.18	.01	**.064**
	.035	.18	.01	**.071**

Fig. 5—Oxygen Contents of Rim and Core of Billet Shown in Fig. 6.

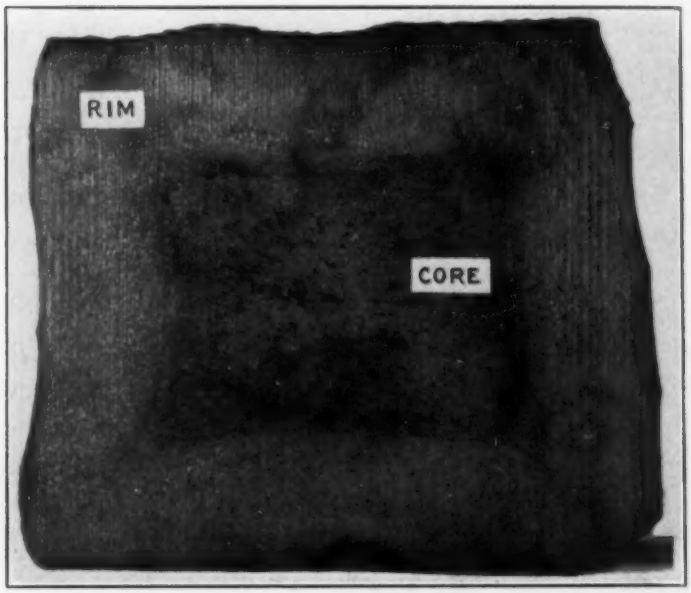


Fig. 6—Cross Section of Billet, from Ingot of Effervescing (rimming, open) Steel.

of Standards,[12] and was devised by them to secure data of the kind we now seek. It is shown in the next slide (Fig. 7). It is 1½ inches square by 2 inches high, which is a size block which upon quenching will harden all over, that is to above 60-C Rockwell, if the steel has a good tendency to harden, like a normal high-carbon steel, and the quenching is fairly drastic, as in cold water. If the steel has any liability to *fail* to harden properly, then soft spots are likely to be found on the block after the quench, that is to say, there are likely to be some areas which after quenching are well below 60-C Rockwell in hardness.

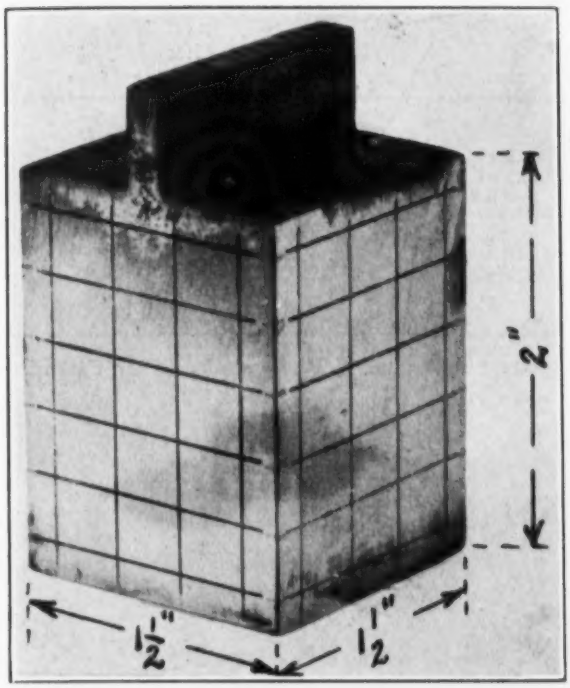


Fig. 7—Block for Making Systematic Investigation of Hardness after Quenching (Rawdon and Epstein).

The examination for hardness is made systematic by drawing a system of vertical and horizontal lines on each face of the block, as shown in the slide, and taking a hardness reading at each intersection. In these tests there were 24 readings on each face.

In proposing to use these blocks for testing the rim and core materials, we recall that the carbon in these materials is very low, about 0.03 per cent, which is of course much too low to give any great hardness upon quenching. We consequently propose carburizing the blocks so as to have high-carbon faces which are therefore hardenable. This may be condoned because a number of the subsequent experiments to be described here have to do likewise with carburized steels.

Before proceeding to the hardness tests after carburizing, we must remember that it is the oxygen content whose effect we are studying, and that the oxygen content changes during carburizing. We must therefore analyze the rim and core materials *after* carburizing. Thin pieces were carburized, ⅛ inch thick, so that they would carburize all the way through, for it seems probable that the oxygen content of these thin pieces after carburizing is a good approximation

	Before carburizing	After carburizing	
Rim	.017 .020	.047 .044	Rim
Core	.064 .071	.094 .089	Core

Fig. 8—Oxygen Content of Rim and Core after Carburizing.

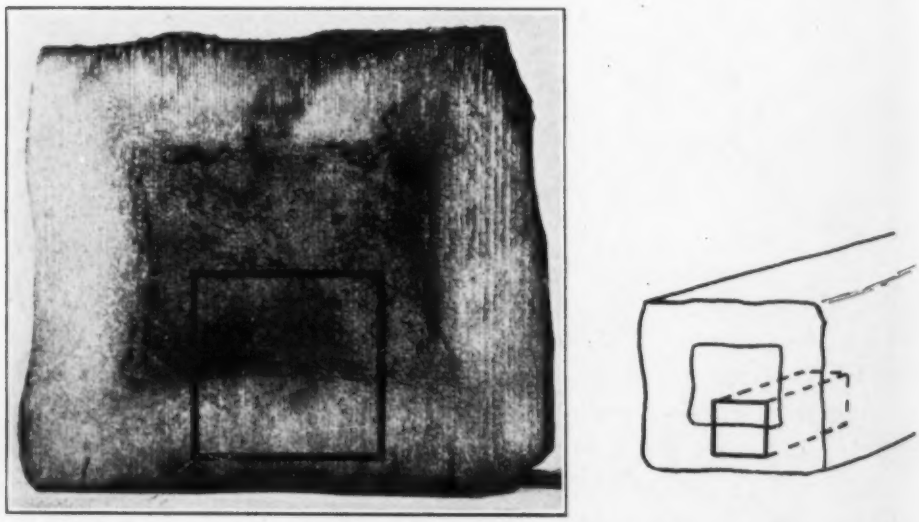


Fig. 9—Manner of Cutting Hardness-testing Blocks from Rim-and-Core Billet.

to the oxygen content of the carburized surface layers of the blocks we shall test for hardness.

The oxygen analyses on the thin pieces are given in the next slide (Fig. 8), and it will be seen that both rim and core increased in oxygen content, and that the core pieces carburized still have a very much higher oxygen content than the rim pieces.

The test blocks could now be prepared for the hardness testing. The manner of cutting the blocks from the rim-and-core billet is shown in the next slide (Fig. 9). Each block was cut so as to be about half rim and half core, since this would show rather strikingly in the same block any differences between rim and core.

The blocks were carburized (8½ hours at 1695 degrees Fahr. (925 degrees Cent.)), and quenched directly from the pot into still water at 65 degrees Fahr. (18 degrees Cent.), and were then examined for hardness. This hardness examination consisted, as was

58–64–59–63	63–60–63–63	64–63–49–63	64–61–64–53
57–56–57–63	63–56–61–64	65–50–44–64	65–44–48–58
64–50–52–64	64–63–63–64	64–53–53–66	64–52–56–59
62–59–51–63	63–64–59–64	64–48–45–65	65–48–54–64
64–64–64–63	63–64–64–64	64–49–65–65	65–65–65–65
63–64–63–63	63–63–63–63	64–65–64–65	66–64–64–63

Fig. 10—Rockwell C Hardness Readings on the Four Faces of a Test Block such as Fig. 7.

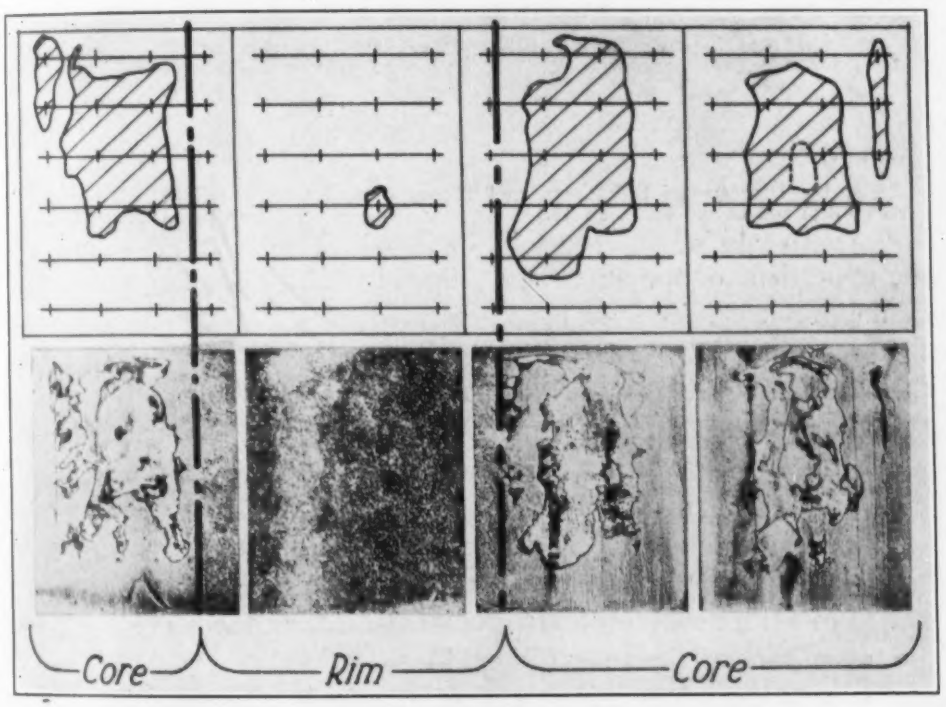


Fig. 11—Diagrams prepared from data of Fig. 10, and actual photographs of the etched faces of the same block.

stated before, in taking 24 hardness readings on each face, at regularly arranged points. The hardness readings for the four faces of a block were listed as shown in the next slide (Fig. 10). From these lists of figures, corresponding diagrams were constructed as shown in

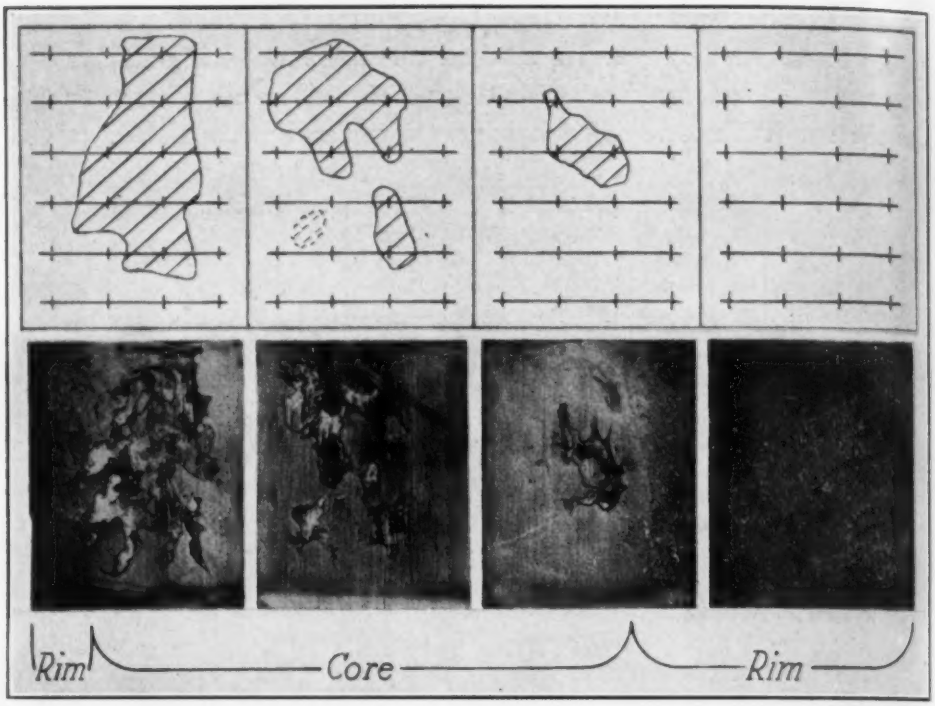


Fig. 12—Diagrams of Soft Spots, and Photographs of Corresponding Etched Faces, in Steel of Rim and Core. (Cf. Fig. 11.)

the upper half of the next slide (Fig. 11). In this diagram the data of the previous slide have been presented so as to show the hard and soft regions. The cross-hatched areas are regions in which all of the hardness readings were less than 60-C Rockwell. In other words, the cross-hatched areas are soft spots, whereas the remaining light areas are regions which were above 60-C Rockwell (i. e. file-hard).

Another method of locating soft spots is to etch the blocks in acid, since the difference in etching between the soft spots and the hard parts is very marked. The block under discussion here was etched in acid, and the photographs of the four faces are shown in the lower half of the slide (Fig. 11).

It will be observed that one of the faces, the second counting from the left, is practically free of soft spots, this behavior extending a short distance along the two adjacent faces, and we note that this is brought out both by the hardness testing and by the etching. This hard portion was the rim portion of the block, i. e. the portion lower in oxygen. The rest of the block, the core portion, showed large soft spots, and the proneness to having soft spots seems attributable to the larger amount of oxygen present in this core portion.

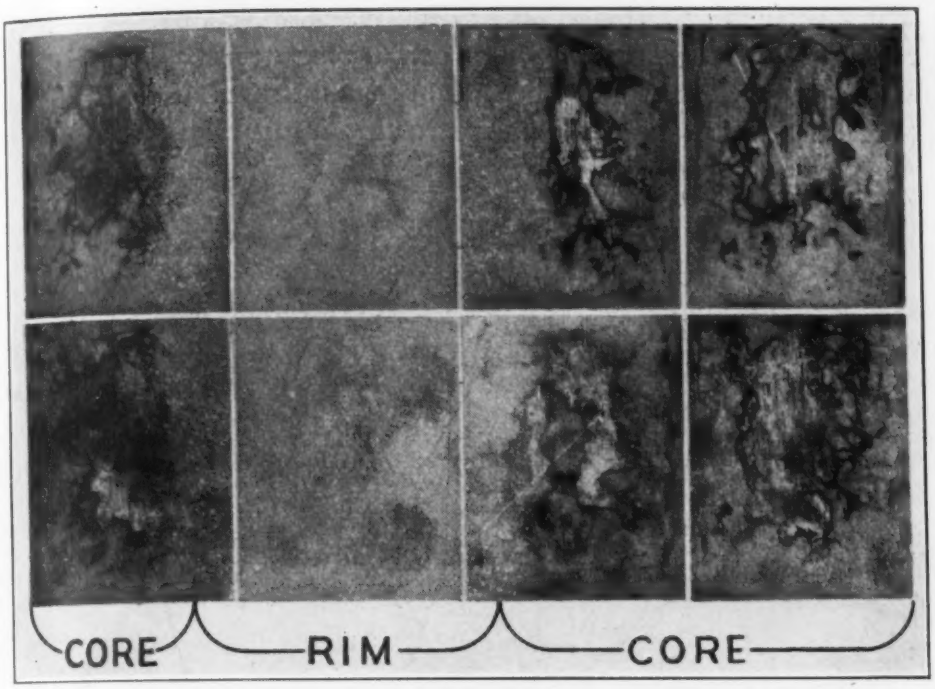


Fig. 13—Etched Faces of Rim-and-Core Blocks. (Cf. Figs. 11 and 12.)

The next slide (Fig. 12) is another block of the same material, and shows the same phenomenon. The face free of soft spots is in this case No. 4, the one at the right. The next slide (Fig. 13) is given merely to show two more examples, the rim portion in each of these cases being at the second face from the left.

* * *

Now up to this point, after having mentioned briefly the amounts of oxygen that are commonly found in steels, we have considered a little evidence that oxygen may sometimes be in the dissolved form, and also some evidence that oxygen may at times affect profoundly the hardening power of a steel in which it is contained.

We come now to what has seemed to be a critical group of experiments, indicating that oxygen in the dissolved form takes on the proportions of a new monster just rearing its head above the metallurgical horizon! As a matter of fact, certain interesting behaviors in steel *can* seemingly be attributed to this new bogie.

We revert here to the classical experiments of McQuaid and Ehn,[13] who discovered a relationship between the tendencies of steel to form soft spots instead of hardening in quenching, and the micro-

structures exhibited by those steels after slow cooling. Their investigations were all carried out on carburizing steels.

An important aspect of their work is illustrated in the next slide (Fig. 14), which shows the microstructure in the high-carbon hypereutectoid zone of two steels which were carburized and then cooled



Fig. 14—Hypereutectoid Zones of Carburized Steels, "normal" (left) and "abnormal" (right), Illustrating McQuaid-Ehn Test. Photomicrographs × 1000.

slowly in the carburizing pot. The outstanding difference between the two is that in the so-called normal steel, the one on the left, the structure consists of grains of pearlite, which are bounded sharply by layers of cementite, whereas in the so-called abnormal sample on the right there are large areas of free ferrite between the pearlite and the boundary cementite. We have, as you see, subscribed to the use of the terms "normal" and "abnormal", not that we wish to enter the controversy as to the suitability of the terms, but merely as a convenience because the terms are after all well established.

McQuaid and Ehn[13] also pointed out that there were marked differences in grain size to be observed in such steels, and indicated that free ferrite and small grain size constituted the features of abnormal steel, which failed to harden properly (that is, the steel showed a tendency to have soft spots). They came to the conclusion that oxides in the steel were responsible for the abnormal condition.

I should like to quote here a comment by the man whose memory we are honoring this morning. It is a letter written to Ehn.

kindly called to my attention by Mr. E. R. Johnson, a former pupil and assistant of Prof. Campbell's, and discusses a possible role of oxygen in the abnormal steels. It reads as follows:

"In normal steel above the critical point the carbides form a homogeneous, probably colloidal solution, which persists until the critical point is reached, when, as the gamma iron goes over to the alpha state, in which the carbides are but slightly soluble, the precipitated carbides assume the form of thin plates, characteristic of normal pearlite. It is well known that the addition of a comparatively small amount of an electrolyte to any colloidal solution in water will bring about coagulation or flocculation of the colloid, and I think it not improbable that the dissolved oxides in your abnormal steels function in a manner exactly analogous to that of electrolytes in aqueous solutions, thus bringing about the flocculation or coalescence of the carbides, either before transformation takes place or during the critical period."

Prof. Campbell's insight here again calls for admiration, since it enabled him to discuss the probable role played by an element which at that time was little understood.

Reference is in order here to publications by Gat,[14] by Inouye[15] and by Harder.[16] Gat stated that the two effects, free ferrite and small grain size, appeared to have different causes, and that it was the condition showing free ferrite, as exhibited in the slide (Fig. 14), that was due to high oxygen, and caused the propensity to show soft spots. Inouye made a series of steels of various oxygen contents, and showed that those higher in oxygen had a greater tendency to show free ferrite after carburizing. Harder examined extensively the amount and mode of formation of hypereutectoid cementite.

A suggestion by Merica resulted in our obtaining some evidence presented on a previous occasion[10] indicating that the free ferrite observable in the slide (Fig. 14) was an area which had been occupied by pearlite, and that the cementite from this pearlite had diffused out to the boundary hypereutectoid cementite and had deposited upon it, making the boundary cementite thicker and leaving free of cementite the area of ferrite which is to be seen here. We shall offer some evidence a little later, which harmonizes with this view that the free ferrite in abnormal steels is a result of the cementite diffusing away from the pearlite of which it was a part.

But if the pearlite in the *abnormal* steel loses its cementite by diffusion out to the boundary cementite, one might reasonably suspect that there is a tendency for such diffusion to take place not only in

abnormal steels, but even in normal steels, and that something conspires to defeat it in the normal steel. Now we know that in the abnormal steel, if the sample is cooled quickly from the carburizing, say in air, instead of slowly as in the carburizing pot, the amount

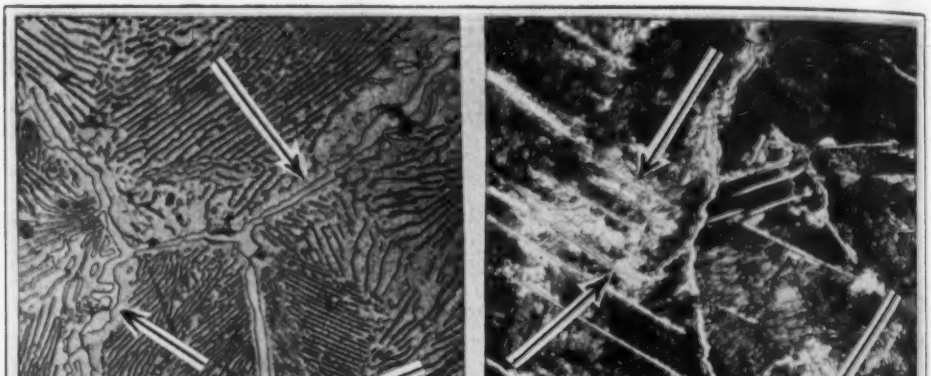


Fig. 15—Normal and Abnormal Steels Cooled to Show Equal Amounts of Ferrite. Normal Steel (left) Cooled in 5000 Minutes, Showed Approximately Same Amount of Free Ferrite as Abnormal Steel (right) Cooled in 1 Minute. Photomicrographs × 1000.

of free ferrite which we find in the structure is then much less, as has been shown by Epstein,[17] that is,—the pearlite is not decomposed so much by diffusion. In other words, by decreasing the time of cooling, during which the separation (the diffusion) takes place, we decrease the amount of free ferrite in the abnormal steel;—and consequently, by increasing the time, we increase the amount. Reverting now to the *normal* steel, if this steel too has a tendency, even though slight, to permit diffusion to take place, then perhaps we could, by prolonging the time of cooling, create conditions which would allow the diffusion of cementite and the occurrence of free ferrite, even in this normal steel.

Experiments were made using various cooling periods. The samples in the slide (Fig. 14) had been cooled in about 45 minutes, this representing the time from carburizing temperature to so low a temperature, say 500 degrees Fahr., that no further reactions could take place. The time of cooling of the normal steel was now successively increased, until, having increased the time of cooling from

45 minutes to about 5000 minutes, the structure in the next slide (Fig. 15) left-hand side, was obtained in the normal steel. It will be seen that diffusion has indeed taken place, and that there are layers of free ferrite, thin but appreciable, indicated by arrows, around the boundary cementite. Now it will be noticed that these layers of free ferrite are very much thinner than those which will be remembered in the abnormal steel which was shown on the right in the previous slide (Fig. 14). So experiments were made with more rapid cooling of the abnormal steel to see if an amount of free ferrite could be obtained which would be roughly the same as that now showing in the normal steel. This was obtained by reducing the cooling time from 45 minutes to about 1 minute, giving the structure illustrated in this slide on the right, indicated by arrows. To repeat, these two steels have such extraordinary differences in rate of diffusion that the normal steel has to be cooled over a period of about 5000 minutes, to show the same amount of free ferrite as develops in the abnormal steel in 1 minute.

This is an extraordinary difference in rate of diffusion, and merits some attention. What could cause such a great difference in diffusion? Obviously something impedes the diffusion of the carbon atoms in one case, or aids their diffusion in the other.

Of course by this time you are already expecting me to say that the difference is due to dissolved oxygen. And I shall not disappoint you—indeed I think so. But the problem is more complex. Why should there be a large amount of dissolved oxygen in some cases, and but a small amount of dissolved oxygen in others.

Now the reasoning at this point was as follows: [If there are large amounts of dissolved oxygen in some cases, and but small amounts in others, then possibly something in the one steel promotes the solution of oxygen, or in another steel obstructs the solution of oxygen. In other words, we have first a difference in the rate of diffusion of carbon, then we attribute this to a difference in amount of dissolved oxygen, and now we are seeking something to which we can attribute the difference in oxygen.]

Now it is well known that steels very high in manganese are usually normal, whereas steels very low in manganese are frequently abnormal. In seeking something to which to attribute a difference in solubility of oxygen, we might therefore look to manganese as a cause. It would seem possible that manganese obstructs the dissolv-

ing of oxygen, with the result that when manganese is present and the dissolved oxygen consequently low, the diffusion of carbide is slow, this being a reason for high manganese steels being normal.

At this point we recall that steels absorb oxygen during carburizing. Now if manganese, as we postulated, obstructs the solution of

Table IV

Change in Oxygen in Pack-Carburizing

Heat No.	C	Mn	Si	Oxygen Before		Oxygen After		Absorbed
668	0.155	0.11	0.09	0.0326	0.0354	0.0708	0.0748	0.039
659	0.16	0.20	0.12	0.0141	0.0158	0.0543	0.0613	0.043
660	0.175	0.20	0.14	0.0124	0.0142	0.0442	0.0482	0.033
661	0.15	0.23	0.11	0.0101	0.0098	0.0778	0.0843	0.071
662	0.16	0.31	0.14	0.0250	0.0238	0.0382	0.0398	0.015
665	0.125	0.32	0.06	0.0290	0.0268	0.0367	0.0344	0.008
663	0.18	0.35	0.12	0.0432	0.0497	0.0552	0.0504	0.006
664	0.23	0.39	0.20	0.0332	0.0333	0.0476	0.0436	0.012
666	0.18	0.50	0.15	0.0264	0.0228	0.0400	0.0445	0.018
667	0.175	0.57	0.18	0.0278	0.0233	0.0385	0.0401	0.014

Steel No.	Mn	Oxygen Before	After	Absorbed
668	.11	.034	.073	.039
659	.20	.015	.058	.043
664	.39	.033	.046	.013
667	.57	.026	.039	.013

Fig. 16—Relation of Manganese Content to Oxygen Absorbed in Pack Carburizing (see Table IV).

oxygen, then possibly high-manganese steels would absorb but little oxygen in carburizing, whereas low-manganese steels might absorb large amounts.

This reasoning led to some successful experiments,—if the experiments had not turned out as predicted we should of course not have mentioned them here. But we do as a matter of fact find, as we shall see in the figures about to be presented, that in general steels low in manganese absorb large amounts of oxygen, whereas steels high in manganese absorb relatively small amounts of oxygen, in carburizing.

An appended table (Table IV) lists ten experimental heats of steel, all of a low carbon carburizing type, which had various manganese contents from 0.11 to 0.57 per cent. The steels were other-

wise quite alike, i. e., aside from their manganese content. These ten heats were pack-carburized (in a regular commercial carburizer of the charcoal type), and analyzed as to oxygen contents before carburizing and after. From the figures given in Table IV, typical values are shown in the next slide (Fig. 16). In the particular instances shown in the slide we note that the two steels relatively low

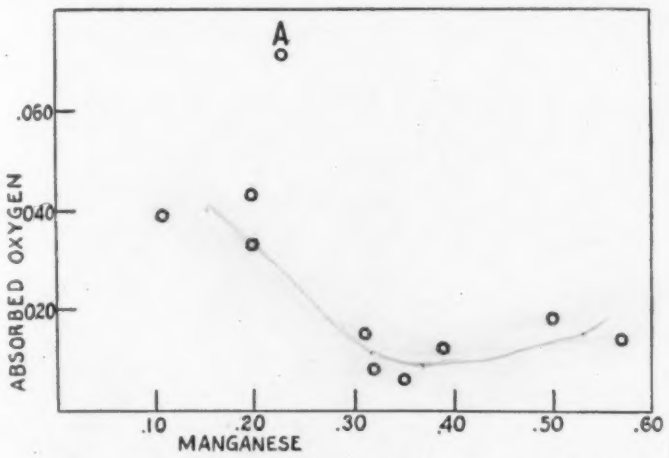


Fig. 17—Relation of Oxygen Absorbed in Pack Carburizing to Manganese Content.

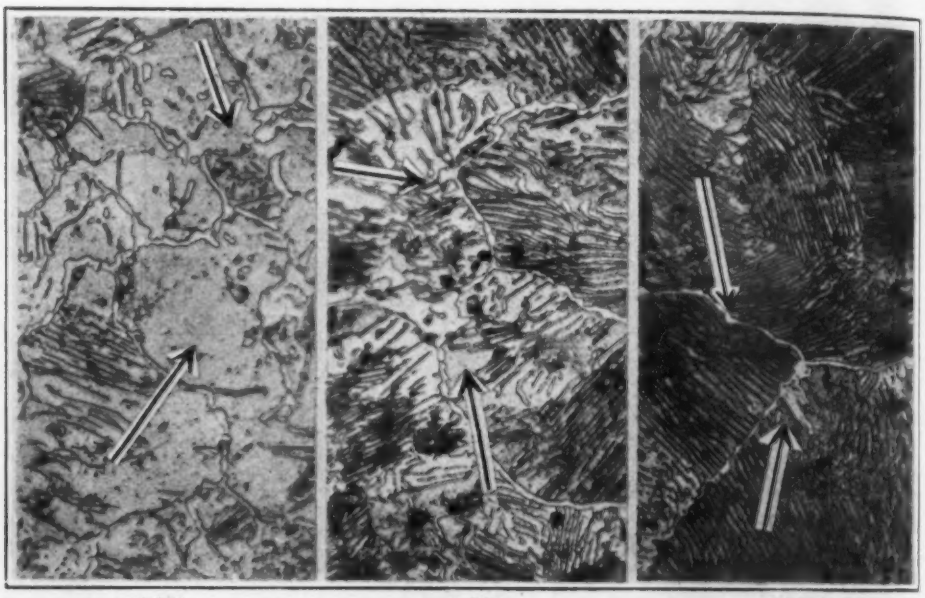
in manganese, i. e., 0.11 and 0.20 per cent manganese, absorbed roughly three times as much oxygen as those having the higher manganese, i. e., 0.39 and 0.57 per cent manganese.

The next slide (Fig. 17) shows in a diagram the relation between the manganese content and the amount of oxygen absorbed in carburizing, for the ten steels mentioned above. We see here the illustration of our general proposition that the steels low in manganese (at the left of the diagram) absorb higher amounts of oxygen than do those containing more manganese shown at the right of the diagram.

The diagram shows also, however, that this relationship between manganese and oxygen is not by any means perfectly regular. It is not only that slight increases in manganese sometimes fail to cause a decrease in absorbed oxygen, but in one case even, designated by the letter A in the diagram, we see that a steel of intermediate manganese absorbed very much more oxygen than any other steel either lower or higher in manganese. We can only conclude from this that, whereas we believe we have discovered one factor which influences the absorption of oxygen in carburizing, there must be other factors yet to be discovered.

We have to content ourselves at present, then, with the knowledge that manganese is one of the factors affecting oxygen absorption. But regardless of the reasons why oxygen is absorbed, once it is absorbed it has some interesting effects on the structure, and it is these effects that we propose to discuss now, examining them in some detail as the concluding chapter of our talk.

The ten steels just described prove upon examination to form an instructive group. If after carburizing we examine the steels



0.071 0.039 0.006

Change in Oxygen Content (Pack Carburizing)

Fig. 18—Hypereutectoid Zones of Carburized Steels, Showing How Amount of Free Ferrite Varies with Amount of Oxygen Absorbed in Carburizing. Photomicrographs × 1000.

under the microscope, using for the sake of control the ends of the pieces that were actually analyzed for oxygen, we find a pronounced trend in the degree of abnormality, that is to say, in the amount of free ferrite. This trend, which seems to us worthy of some attention, is expressed by saying that it is the amount of oxygen absorbed in carburizing that determines the degree of abnormality,—that is to say, the amount of free ferrite is dependent not on the total oxygen content of the steel but apparently almost solely on the amount of oxygen absorbed in carburizing. This is illustrated in the next slide (Fig. 18) which shows three of the steels, the one which absorbed the most oxygen (0.071 per cent) at the left, then an intermediate one

(0.039), and then the least (0.006) at the right. The arrows point to the significant areas. We see in these carburized zones, where oxygen was absorbed as well as carbon, that the steel absorbing large amounts of oxygen then developed much free ferrite, as shown on the left, that intermediate oxygen absorption led to a lesser amount of free ferrite, and finally, that the steel which absorbed no appreciable amount of oxygen in carburizing developed likewise no appreciable amount of free ferrite, as shown on the right. These three photographs are typical of the conditions found in the ten steels.

This evidence makes it seem probable that it is the oxygen absorbed in carburizing which is in general responsible for the abnormality of steel. This would seem to be true at least for the carburizing steels in general use, because the other oxygen, the oxygen already in the steel, which remains as a residue from the melting processes, is in a form which is not readily dissolved at steel treating temperatures. *That* oxygen is present largely as inclusions of the silicate type, because the steel is usually a so-called killed steel, containing appreciable amounts of silicon. The ten steels with which we have just experimented were of that type. It should be remembered, however, that other kinds of steels may be encountered. Indeed you may recall that one of the steels discussed this morning was an effervescing or "open" steel, in which we studied the rim and the core (Figs. 5 to 12). In such steels it appears that some of the oxygen originally present in the steel may be quite soluble at steel-treating temperatures, and would therefore take part in the reactions under discussion.

But let us return to our statement that it is usually only the oxygen absorbed in carburizing that leads to free ferrite and causes abnormality. At this point we are in hopes that you desire a little further evidence, for the very good reason that we are about to offer you some. And our procedure is this: if it is absorbed oxygen that causes abnormality, then if a steel could be carburized without absorbing oxygen, it should remain normal and show no free ferrite.

Such carburization, where the absorption of oxygen was largely avoided, was carried out in this case by carburizing with methane CH_4 gas. We carburized again the ten steels discussed before, and, bearing in mind the researches of Sykes,[18] Guthrie[19] and others, did the carburizing with methane derived from natural gas. An appended table (Table V) gives the oxygen analyses before and after carburizing, and we find that, whereas in pack or CO carburizing, the greatest

Table V
Change in Oxygen in Gas-Carburizing

Heat No.	C	Mn	Si	Oxygen Average before	Oxygen After	Change
668	0.155	0.11	0.09	0.0340	0.0309	−0.003
659	0.16	0.20	0.12	0.0150	0.0263	+0.011
660	0.175	0.20	0.14	0.0133	0.0257	+0.012
661	0.15	0.23	0.11	0.0100	0.0122	+0.002
662	0.16	0.31	0.14	0.0244	0.0332	+0.009
665	0.125	0.32	0.06	0.0279	0.0223	−0.006
663	0.18	0.35	0.12	0.0465	0.0380	−0.009
664	0.23	0.39	0.20	0.0333	0.0332	0
666	0.18	0.50	0.15	0.0246	0.0251	+0.001
667	0.175	0.57	0.18	0.0256	0.0264	0

gain in oxygen reached as much as 0.071 per cent, in methane CH_4 carburizing the greatest gain was only 0.013. That there was any gain at all is probably an index that the gas was not quite pure.

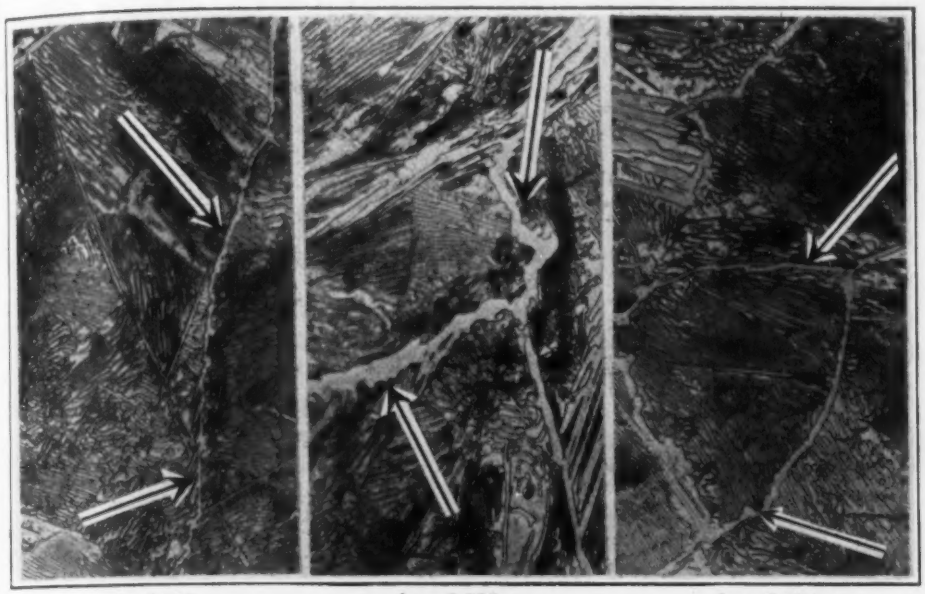
For comparison with an earlier slide (Fig. 16) where we showed gains in oxygen varying from 0.013 up to 0.043, we show in the next

Steel No.	Mn	Change in Oxygen Pack, CO	Methane, CH_4
668	.11	+.039	−.003
659	.20	+.043	+.011
664	.39	+.013	0
667	.57	+.013	+.001

Fig. 19—Table Showing that Pack Carburizing (left) Leads to much Greater Oxygen Absorption than Methane Carburizing (right). (See Tables IV and V.)

slide (Fig 19) the same four steels carburized under the new conditions. Here the changes in oxygen ranged from a loss of 0.003 in one case to a gain of 0.011 per cent in another case. Altogether, we find the changes in oxygen content to be very slight indeed.

And now we come to the structures under the microscope. We find that when the steels are carburized with methane CH_4 to avoid oxygen absorption, none of them show free ferrite or abnormality to any appreciable extent, in other words the steels have remained quite normal. We showed in an earlier slide (Fig. 18) three of the steels carburized under the previous conditions, namely one which absorbed a very large amount of oxygen and was quite abnormal, one which absorbed an intermediate amount and was somewhat abnormal, and



gain 0.002 loss 0.003 loss 0.008

Change in Oxygen Content (Using Methane)

Fig. 20—Steels Carburized in Methane. Change in Oxygen Content Negligible. Steels Normal. Photomicrographs × 1000.

one which absorbed practically no oxygen and remained normal. In the next slide (Fig. 20) we show these same three steels, carburized under the new conditions where there was practically no absorption of oxygen. The steel which had absorbed 0.071 oxygen and had become quite abnormal, now absorbed only 0.002 and remained normal, as shown on the left. The intermediate one which had absorbed 0.039 and had become somewhat abnormal, now *lost* 0.003 and remained normal. The one on the right which before had gained only 0.006 per cent oxygen and which was normal in the previous case also, now lost 0.008 per cent and was again normal.

To recall the structures to mind, we show in the next slide (Fig. 21) the steel which had absorbed the most oxygen and had become the most abnormal, shown on the left, and compared with it on the right the same steel carburized under the new conditions, where no appreciable oxygen was absorbed, the steel remaining normal. The arrows point as usual to the significant areas.

We find, then, distinct evidence in these ten steels that the degree of abnormality or amount of free ferrite was proportional to the amount of oxygen absorbed in pack carburizing, and that when the absorption of oxygen was prevented, the steels had a normal structure.

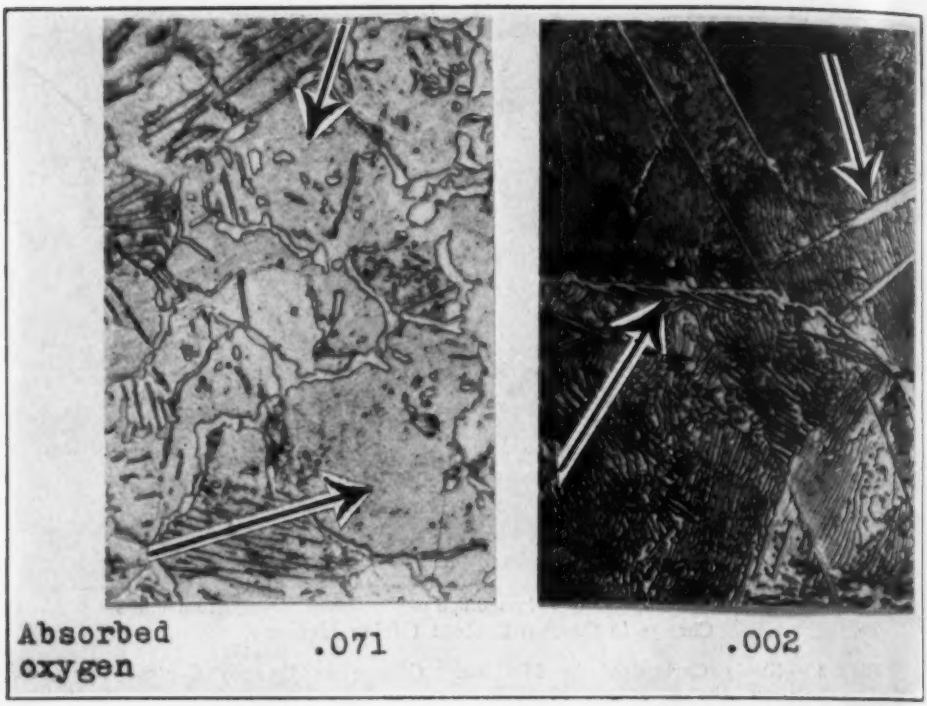


Fig. 21—Steel which Absorbed 0.071 Oxygen in Pack Carburizing and Became Abnormal (left), Absorbed 0.002 Oxygen in Methane Carburizing and Remained Normal (right). Photomicrographs × 1000.

And now a final word as to this normal structure. We have talked very glibly all morning about speed of diffusion of the carbide, saying that when dissolved oxygen was present, the carbide diffused faster. That is, in the presence of dissolved oxygen, the carbide of the pearlite diffused out and deposited on the hypereutectoid cementite at the boundaries of the grains, leaving free ferrite, but when dissolved oxygen was *not* present, the diffusion was so slow that the carbide was unable to migrate sufficiently and the structure remained normal. But if these differences in diffusion are found *within* the steels, we might reasonably look for differences also at the surfaces of the test pieces,—that is, we might expect to find differences in the rate at which carbon diffuses into steel or diffuses out of it—i. e., differences in the rate of carburization or decarburization.

Now while the structure by which we judge abnormality under the microscope, is formed during the cooling of the test piece after carburizing, it has been our belief[10] that this structure is formed in the temperature range just *below* the critical point, that is, *after* the transformation from austenite to ferrite. That is to say, although

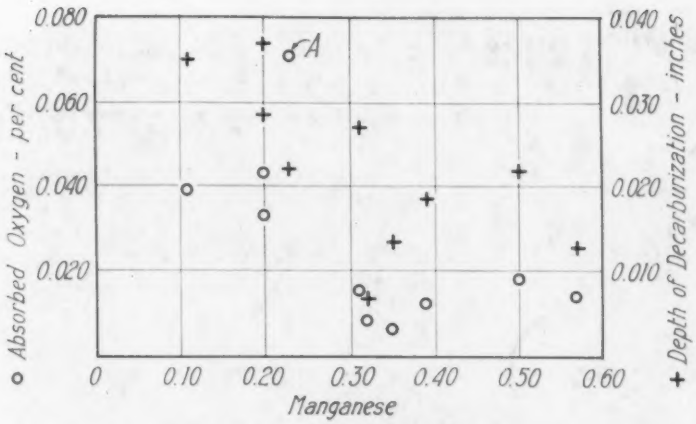


Fig. 22—Relation of Depth of Decarburization at 1250 Degrees Fahr. (675 Degrees Cent.) to Manganese Content, Plotted on Same Chart as Fig. 17, which Showed Oxygen Absorption.

the oxygen and the carbon are absorbed while the steel is austenite, the peculiar diffusion of the *carbide* which causes abnormality takes place only after the transformation to ferrite. The evidence here has to do with a point brought out in conversation with Mr. E. C. Bain and again with Dr. R. H. Aborn. It is as follows: We know that the speeds of carburization of normal and abnormal steels are about alike,—that is, if a piece of normal and a piece of abnormal steel are carburized under the same conditions, the depth of carburization is likely to be about the same in the two pieces, differing usually by not more than one-tenth of the total, or at most one-fifth. This indicates that the speeds of diffusion *above* the critical point, i. e., during carburizing, are quite similar. But if the differences in diffusion in which we are interested occur *below* the critical point, then we ought to carry out the tests at that lower temperature and look for differences there.

The same ten steels were therefore now packed in carburizing compound, and were heated at 1250 degrees Fahr. (675 degrees Cent.) for about 24 hours. At this low temperature the atmosphere in the mixture is decarburizing as to these steels, so that we find the surface layers not carburized but decarburized. And we are struck at once by the very great differences in extent of decarburization. Whereas *above* the critical point the diffusion of carbon into the steels differed by not more than one-fifth of the total, here *below* the critical point we find the carbon diffuses out of the steel to a depth which in some cases is 4 to 6 *times* as much as in others. And we find, as illustrated in the next slide (Fig. 22) that the steels low in

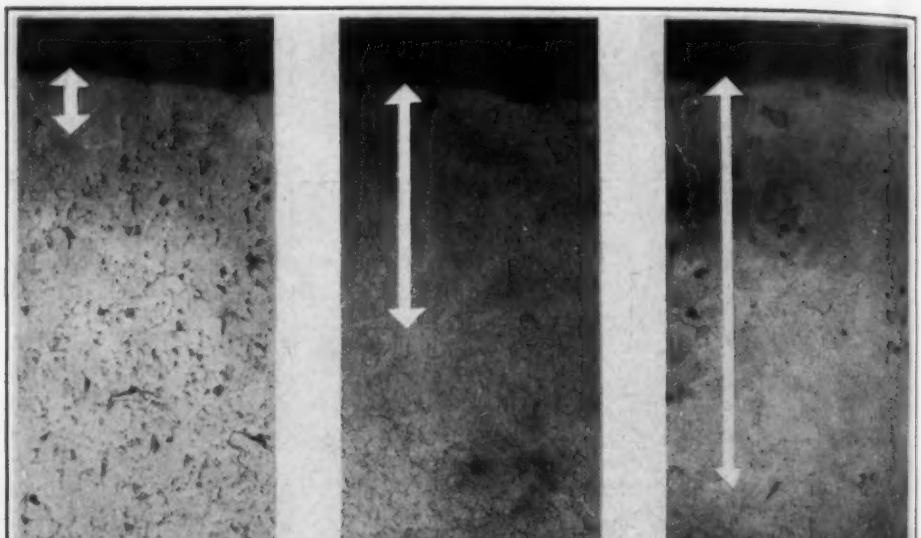


Fig. 23—Decarburized Surface Layers of Steels Heated at 1250 Degrees Fahr. (675 Degrees Cent.). Photomicrographs × 100.

manganese decarburized the most, these being the same steels at the left of the diagram which in general absorbed the most oxygen, and were the most abnormal. The crosses in this slide indicate depth of decarburization, the circles indicating as in a previous slide (Fig. 17) the amount of oxygen absorbed in carburizing.

The next slide (Fig. 23) shows a few typical microstructures from which the depths of decarburization were estimated, the arrows showing extent of decarburized material. Now whether this decarburization is again dependent on oxygen absorbed during this same treatment, or on some other factor, we are as yet without evidence. What *does* appear, is that the carbide diffused below the critical temperature the fastest in the most abnormal steels.

In concluding, it is pleasant to acknowledge publicly my indebtedness to three individuals; first Mr. Frederick J. Griffiths, whom I affectionately term "the boss," and who by his encouragement has helped more than perhaps anyone else; then I should like to mention Mr. J. C. Joublanc who personally made all of the analytical determinations of oxygen reported here; and finally the chairman of this meeting, my friend and counselor, Prof. Albert Sauveur, in whose laboratories a great many of these experiments were carried out, and to whom I feel deeply indebted for his kindness.

Bibliography

1. Ledebur, Stahl und Eisen, Vol. 2, 1883, p. 502.

2. Oberhoffer, P., Rev. Techn. Luxbg., 1927, p. 39 ff.

3. Wasmuht, Roland and Oberhoffer, Paul, Ueber die Bestimmung der oxydischen Einschlüsse in Eisen und Stahl auf rückstandsanalytischem Wege durch Chloraufschluss, *Archiv für das Eisenhüttenwesen,* June 1929, p. 829.

4. Dickenson, Distribution of Silicates in Steel Ingots, *Journal,* Iron and Steel Institute, Vol. 113, 1926-I, p. 177.

5. Jordan, L., and Eckman, "Gases in Metals," Bureau of Standards. Scientific Paper No. 514.

6. Oberhoffer, P., Schiffler, H. J., and Hessenbruch, W., "Sauerstoff in Eisen und Stahl," *Archiv für das Eisenhüttenwesen,* July 1927, p. 57.

7. Hessenbruch, W., and Oberhoffer, P., "Ein verbessertes Schnellverfahren zur Bestimmung der Gase in Metallen, insbesondere des Sauerstoffs im Stahl," *Archiv für das Eisenhüttenwesen,* March 1928, p. 583.

8. Diergarten, Hans, "Zur Bestimmung der Gase in Metallen, besonders des Sauerstoffs in Eisen und Stahl, nach dem Heissextraktionsverfahren," *Archiv für das Eisenhüttenwesen,* June 1929, p. 813.

9. Diergarten, Hans, "Zur Bestimmung der Gase in Metallen, besonders des Sauerstoffs in Eisen und Stahl, nach dem Heissextraktionsverfahren. II. Teil: Anwendung des Verfahrens; Sauerstoffseigerung; Sauerstoffwert und metallographischer Befund," *Archiv für das Eisenhüttenwesen,* March 1930, p. 577.

10. Grossmann, M. A., "On Oxygen Dissolved in Steel, and its Influence on the Structure." TRANSACTIONS, American Society for Steel Treating, Vol. XVI, July 1929, p. 1.

11. Treinen, Leo, Doctor's Thesis, Technische Hochschule, Aachen, 1929.

12. Epstein and Rawdon, TRANSACTIONS, American Society for Steel Treating, Vol. 12, 1927, p. 337.

13. McQuaid, H. W., and Ehn, E. W., *Transactions,* American Institute of Mining and Metallurgical Engineers, Vol. 67, 1922, pp. 341-91.

14. Gat, J. D., TRANSACTIONS, American Society for Steel Treating, Vol. 12, 1927, pp. 376-435.

15. Inouye, K., *Mem. Col. of Eng. Imp. Univ. Fukuoka,* Japan, Vol. 5, 1928, pp. 1-69.

16. Harder, O. E., Weber, L. J., and Jerabek, T. E., TRANSACTIONS, American Society for Steel Treating, Vol. 13, 1928, pp. 961-1008. Harder, O. E., and Johnson, W. S., TRANSACTIONS, American Society for Steel Treating, Vol. 15, Jan. 1929, p. 49.

17. Epstein, Discussion of paper by Grossmann, reference (10).

18. Sykes, TRANSACTIONS, American Society for Steel Treating, Vol. 12, Nov. 1927, p. 737.

19. Guthrie and Wozasek, "Gas Carburization of Steel," TRANSACTIONS, American Society for Steel Treating, Dec. 1927, p. 853.

CRITICAL RANGES IN PURE IRON-CARBON ALLOYS

The A_3 and A_{cm} Ranges, and the Merging Point of A_3 and A_2.

By R. H. Harrington and W. P. Wood

Abstract

A series of 32 pure iron-carbon alloys was prepared by the use of the method devised by the late Prof. E. D. Campbell. Twenty-two of these steels had been prepared by Prof. Campbell. The carbon contents of these samples were checked by chemical analysis and the steels examined metallographically. Thermal analyses of these steels were made by the use of a modified Rosenhain furnace. The inverse rate method of plotting was used. From the data thus obtained heating and cooling diagrams were drawn and the location of the ranges on the thermal point diagram determined by averaging the temperatures of the heating and cooling diagrams. The difference in temperature between the heating and cooling ranges was about 6 degrees Cent. for the A_3 transformation. Greater differences were noted for the A_2 and A_{cm} ranges.

The A_3 transformation apparently takes place in two steps as shown by a persistent double cusp on the heating and cooling curves. The merging point of A_3 and A_2 was found to occur at 0.60 per cent carbon and 744 degrees Cent. (1370 degrees Fahr.) instead of 0.38 per cent carbon as indicated on previous diagrams. The A_{cm} transformation appears to be more nearly a straight line than a curve as usually shown.

The A_1 and $A_{1\text{-}2\text{-}3}$ ranges appear to involve some new phenomena, the nature of which are not quite clear at the present time. Further work upon these ranges will be presented in a subsequent paper.

1. Introduction

TO one studying the data upon which the present iron-carbon diagram was constructed, it seems that some of the points

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The authors are members of the society. R. H. Harrington is research metallurgist, General Electric Co., Schenectady, N. Y., and W. P. Wood is associate professor of metallurgical engineering, University of Michigan, Ann Arbor, Mich. This paper constitutes a part of a thesis submitted in partial fulfillment of the requirements for the degree of Doctor of Science at the University of Michigan. Manuscript received February 24, 1930.

used were rather uncertain as to location and in some instances comparatively few in number to establish diagram lines. It is also evident that little work on a comprehensive scale has been published since the investigations of Carpenter and Keeling[1] in 1904. Much of the earlier work was carried out on specimens which contained appreciable amounts of manganese, silicon, sulphur and phosphorus, so there was a possible influence upon the points obtained through the presence of these elements.

Prof. E. D. Campbell during the later years of his life had occasion, in the process of his investigations, to utilize steel specimens which contained practically nothing but iron and carbon. He developed a process for the production of such alloys and described it in a paper[2] presented before the sixth annual convention of this society. After his death it was suggested that the samples prepared by him together with others prepared by one of the authors be used in making an exhaustive study of the critical ranges in such alloys, over the range 0 to 1.5 per cent carbon. This work was started and the present paper presents part of the results obtained thus far.

2. Work of Previous Investigators

Prof. Albert Sauveur[3] was one of the first to publish informa- complete iron-carbon series. He plotted the diagram from cooling curves of sixteen iron-carbon alloys, nine of which contained less than 1.7 per cent carbon. Dr. Albert Stanfield[5] published a paper width.

Roberts-Austen[4] was the first to publish the diagram for the complete iron-carbon series. He plotted the diagram from cooling curves of sixteen iron-carbon alloys, nine of which contained less than 1.7 per cent carbon. Dr. Albert Stansfield[5] published a paper

[1]H. C. Carpenter and D. F. E. Keeling, "Range of Solidification and the Critical Ranges of Iron-Carbon Alloys," *Journal* Iron and Steel Institute, Vol. LXV, 1904, p. 224.

[2]E. D. Campbell and G. W. Witney, "A Laboratory Method for the Preparation of Small Steel Bars Differing Only in Carbon Content and the Effect of Changes in Carbide Concentration on the Specific Resistance," Transactions American Society for Steel Treating, Vol. VI, 1924, p. 33.

[3]Albert Sauveur, "The Microstructure of Steel and Current Theories of Hardening," *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol XXVI, 1896, p. 863.

[4]W. C. Roberts-Austen, Fourth Report of the Alloys Research Committee, *Proceedings* Institution of Mechanical Engineers, 1897, pages 70 and 90.

[5]Alfred Stansfield, "Present Position of the Solution Theory of Carburized Iron," *Journal* Iron and Steel Institute, Vol. LVI, 1899, p. 169.

in which he interpreted the Roberts-Austen diagram by means of a "solution theory."

Roozeboom[6] applied the phase rule to the scattered data of Roberts-Austen's cooling curves. The lines on the diagram in Fig. 1 were established by this application of the phase rule to the data of Roberts-Austen. This is essentially the form of the diagram as it has been used since.

In 1904 Carpenter and Keeling[7] ran cooling curves on thirty-eight alloys of iron and carbon. Material from six different sources was used in preparing this series. The alloys were melted in a gas-fired crucible furnace. Eighteen of these alloys were used in determining the position of the critical ranges in steel. The carbon contents varied from 0.01 to 1.69 per cent. The A ranges were determined by means of the differential method, a platinum blank being employed. The heating and cooling of the samples was accomplished in a nickel-wound tubular electric resistance furnace. The points on Fig. 1 were obtained from the cooling curves of Carpenter and Keeling. The results of this research were regarded at the time as sufficient proof of the accuracy of Roozeboom's deductions from the work of Roberts-Austen. This diagram with only minor changes has been used to the present time.

Since 1904 many improvements in the technique of thermal analysis have been made and this led to the belief that a careful repetition of the work of Carpenter and Keeling might yield fruitful results. The points leading to this conclusion are summarized:

1. Possible errors in measurement of temperatures due to use of soldered junctions and the fact that only the thermocouple receiving the most drastic treatment was checked and this only before and after the entire series of curves was taken.
2. Improvements in methods of chemical analysis.
3. Alloys used by Carpenter and Keeling contained appreciable amounts of other elements.
4. The differential method used by Carpenter and Keeling involved the use of a platinum blank placed from one to two inches away from the steel sample in the furnace.
5. The runs of Carpenter and Keeling were made in an atmosphere of air.

[6]H. W. Bakhuis-Roozeboom, "Iron and Steel from the Point of View of the 'phase doctrine,'" *Journal* Iron and Steel Institute, Vol. LVIII, 1900, p. 311.

[7]Refer to footnote 1.

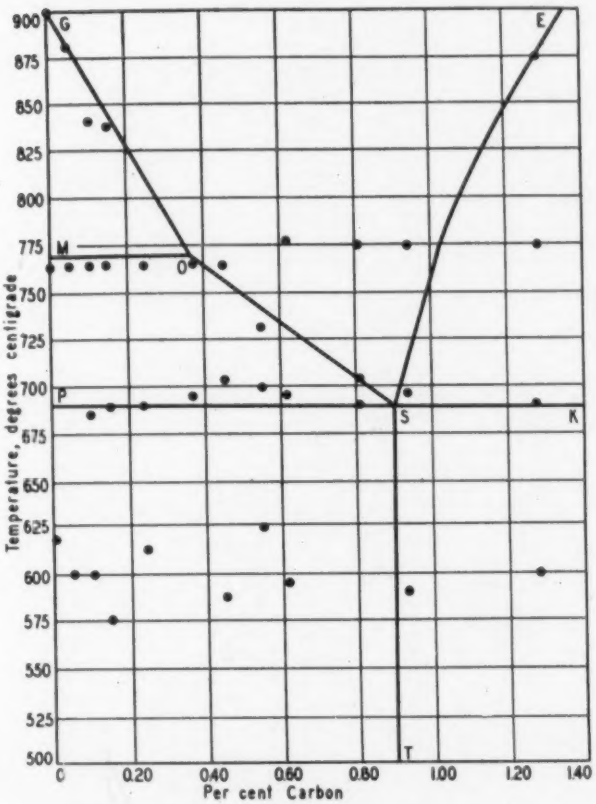


Fig. 1—The Critical Ranges in Steel. The Lines are Taken from the Original Roozeboom Diagram. The Points are Taken from the Cooling Curves of Carpenter and Keeling.

6. Samples used were quite large and possibly involved considerable lag as well as possible uneven distribution of carbon.

7. Apparently no metallographic examination of the samples was made.

8. Only nine curves of the thirty-eight of Carpenter and Keeling were published and there is considerable difficulty in reading an exact temperature from them.

9. Cooling curves obtained by very slow cooling were regarded as representing equilibrium conditions so closely as to allow the points obtained to be plotted as an equilibrium diagram.

10. Some points were obtained for which no explanation was offered.

The work done since 1904, relative to the diagram, ranges from that of Arnold and McWilliams[8] to the recent theoretical con-

[8] John O. Arnold and Andrew McWilliams, "Thermal Transformations in Carbon Steels," *Journal* Iron and Steel Institute, Vol. LVIII, 1905, p. 27.

Table I

Analyses of Armco Irons

Used by Authors	*Used by Prof. E. D. Campbell*
Per cent	Per cent
0.037 Carbon	0.010 Carbon
0.045 Manganese	0.028 Manganese
0.007 Phosphorus	0.003 Phosphorus
0.015 Silicon	Trace Silicon
0.020 Sulphur	0.018 Sulphur
Total impurities = 0.087 per cent.	Total impurities = 0.050 per cent.

siderations of Donovan Jones.[9] Some forty other references have been consulted, but there is not space here to review them. Most of the work published in the past twenty years checks relatively well the excellent work of Carpenter and Keeling. However, some of the data, very carefully obtained and well established, does not seem to agree with the rest. It is in the hope of clarifying the situation and in the nature of a plea for a physicometallic theory that the present work is published.

3. Preparation of the Steels

The method used in preparation of the steel samples is based on the fact that at 960 degrees Cent. (1760 degrees Fahr.) in a constant atmosphere of dry hydrogen, equilibrium between all the iron and all the carbon is reached in six days. Thus, if a bar of 0.60 per cent carbon steel and two bars of pure iron (all the same size) are used in a run, all three bars will have a carbon content of 0.20 per cent when equilibrium is reached. By adjusting the ratio of high carbon bars to low carbon bars, any desired carbon content may be obtained. The high carbon bars are obtained by carburizing part of the original bars up to 1.25 per cent carbon. The carburization and equilibrium runs were made in the furnaces which Professor Campbell originally used for those purposes.

Armco iron, furnished through the courtesy of J. Hunter Nead, was the original material used in preparation of the samples by Professor Campbell and the authors. Table I contains the analyses of the two original Armco irons.

Table II contains the carbon contents of the complete series of thirty-two steels. The steels marked with an asterisk were prepared

[9]Donovan Jones, "An Assumption as to the Cause of the Allotropic Changes of Iron," Transactions American Society for Steel Treating, Vol. XIV, 1928, p. 199.

Table II
Carbon Contents of the 32 Steels

No. of Bar	Per Cent Carbon	No. of Bar	Per Cent Carbon
1	0.000	17*	0.454
2	0.030	18*	0.517
3*	0.037	19	0.590
4	0.054	20*	0.609
5	0.070	21*	0.644
6	0.100	22	0.660
7*	0.153	23	0.740
8	0.160	24*	0.767
9	0.200	25	0.820
10	0.210	26	0.900
11*	0.270	27	0.990
12*	0.370	28	1.110
13	0.390	29	**1.130**
14*	0.400	30	1.121
15	0.430	31	1.480
16	0.440	32	1.500

by the authors, the remaining twenty-two samples having been obtained from Professor Campbell's stock. It may be noted that a very close grouping of the steels exists from 0.37 per cent to 0.45 per cent carbon which includes the merging point of A_3 and A_2 at 0.38 per cent carbon on the Carpenter and Keeling diagram. (Fig. 1).

The carbon contents of these steels were calculated from the differences in weights of the bars before and after the equilibrium runs. These values were checked by the combustion method of carbon determination.

4. Metallographical Examination

Sections of each of the thirty-two steels were taken ½ inch from one end of each bar, polished, etched with 3 per cent nitric plus 3 per cent picric acid in ethyl alcohol, and examined under the microscope at magnifications of 100 and 500. Figs. 2, 3 and 4 present photomicrographs of twenty-four of the steels, eight being omitted as their carbon contents are nearly identical to eight of those presented.

All of the steels showed even distribution of the pearlite. The carbonless iron bar was found to consist almost entirely of one grain, two small grains appearing at the very edge of the sample. There is the expected tendency for the grain size to decrease as the carbon content increases. The photomicrograph of the 0.037 per cent carbon steel shows the small elongated structure of the original rolled plate of Armco iron from which the steels were made.

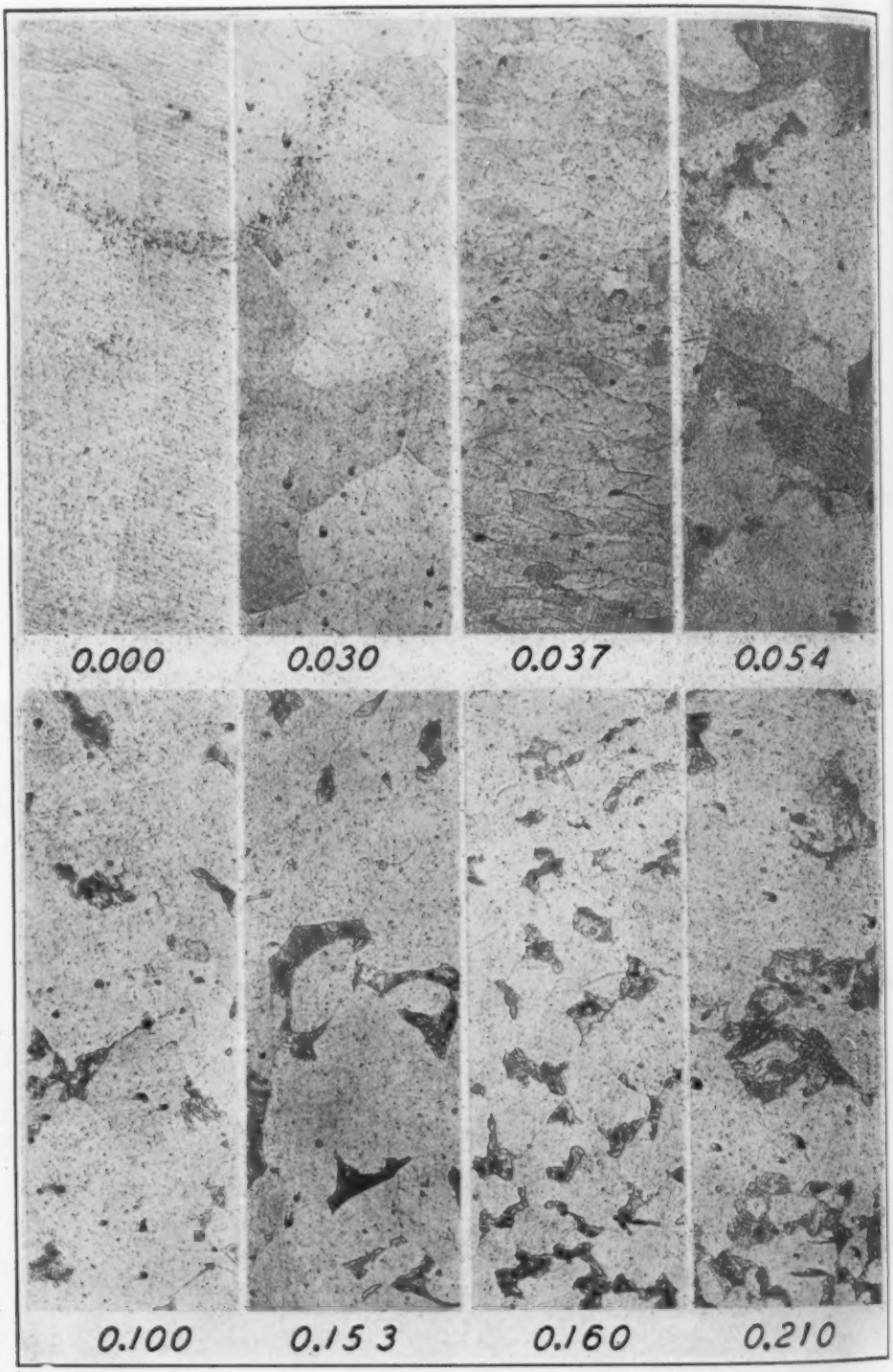


Fig. 2—Photomicrographs of Annealed Steels. Carbon Contents are Indicated Under Each Print. × 100.

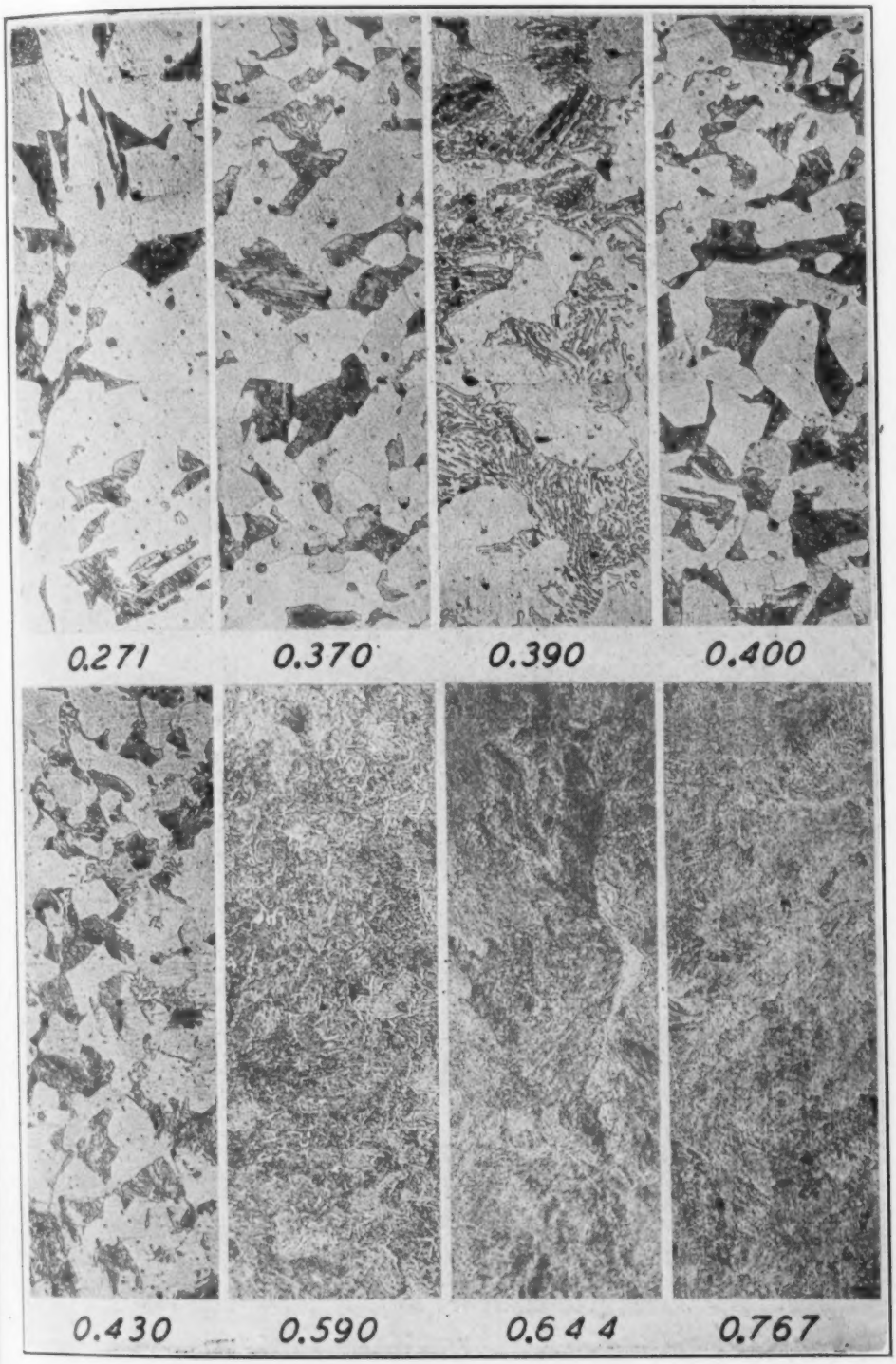


Fig. 3—Photomicrographs of Annealed Steels. Carbon Contents are Indicated Under Each Print. × 100.

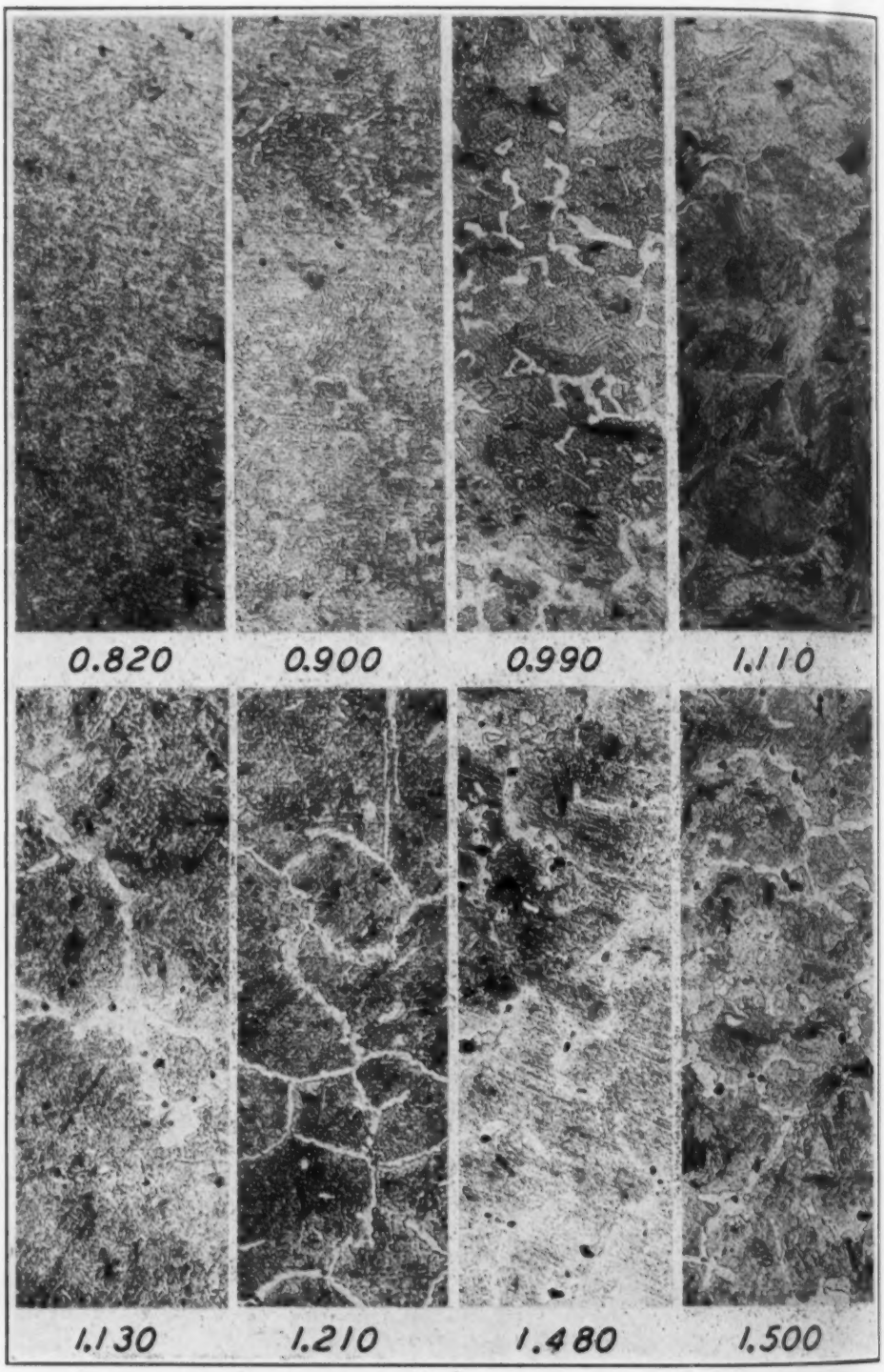


Fig. 4—Photomicrographs of Annealed Steels. Carbon Contents are Indicated Under Each Print. × 100.

The 0.767 per cent carbon steel shows a few small areas of ferrite while the 0.90 per cent carbon shows considerable excess of cementite. The 0.82 per cent carbon steel consists entirely of one

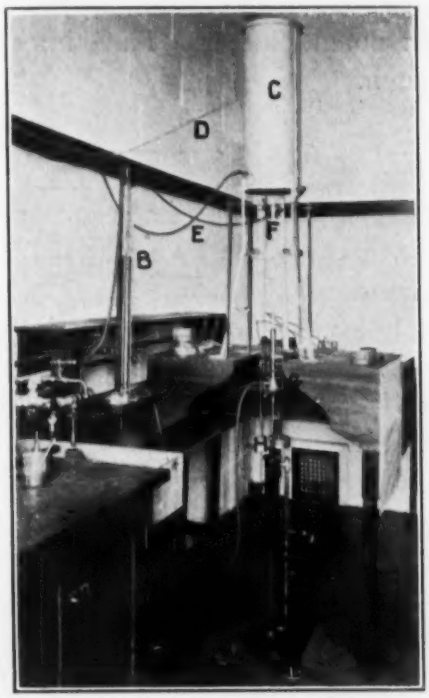


Fig. 5—Photograph of Modified Rosenhain Furnace. (A) Water Aspirator. (B) Manometer. (C) Furnace. (D) Electric Leads to Furnace. (E) Water Leads to Cooling Coil. (F) Pyrometer Tube Containing Sample in Upper End. (G) Vacuum Cup on Traveling Platform. (H) Weight Pans and Lead Weights. (I) Oil Cylinder Containing Elevating Piston. (J) By-pass on Oil Cylinder and Needle Valve to Regulate Rate of Movement of Elevating Mechanism.

constituent, pearlite, and therefore seems to be of eutectoid composition. The hypereutectoid steels are all rather coarse grained.

5. Apparatus for Thermal Analysis

Thermal analyses were carried out in a modified Rosenhain[10] furnace as described by Scott and Freeman.[11] Some minor varia-

[10]Walter Rosenhain, "Some Appliances for Metallographic Research," *Journal* Institute of Metals, Vol. XIII, 1915, p. 160.

[11]H. Scott and J. R. Freeman, "Use of a Modified Rosenhain Furnace for Thermal Analysis," Scientific Paper, No. 348, U. S. Bureau of Standards.

tions in this apparatus were made. Fig. 5 shows the set-up of the furnace and its accessories.

Temperatures were determined by means of a new platinum, platinum-10 per cent rhodium thermocouple, millivoltage being measured by a deflection potentiometer. Time intervals were taken by means of a stop watch.

6. Procedure

Preparation of Samples: The dimensions of the samples were 5/8 by 1/4 by 1/4 inch. A 3/16 inch hole was drilled longitudinally in each sample to a depth of 5/16 inch. By this means, the samples were enabled to fit like a cap over the bead of the thermocouple and extend down over the two-hole insulator for a distance of 1/4 inch. The use of a light oil kept the samples cool while they were drilled. Each sample was washed with alcohol and dried before being used in a run.

Checking of the Thermocouple: The couple used in the furnace was checked against a noble metal couple standardized by the U. S. Bureau of Standards. Checks were made after each six runs. No variations at any time were found.

Details of a Run: After washing with alcohol, the sample was placed over the head of the thermocouple insulator and the pyrometer tube slipped over it and into the sleeves of the vacuum cup. The vacuum cup was then secured on the platform over the oil-cylinder and the wires to the weight pans were attached to the arms on the platform. The water aspirator was turned on and the pressure in the sample tube reduced to a point between 9 and 15 millimeters of mercury, all junctions on the vacuum cup having been sealed with a mixture of beeswax and vaseline. Weights were placed on the weight pans in sufficient quantity to insure an even rate of elevation. The same weights were used on each run. The valve in the by-pass tube had a setting for each heating run such that the speed was one inch in 4½ minutes. While cooling the sample, the weights were hung directly to the arms of the platform and a different setting of the valve was necessary to obtain the same rate of platform travel as during heating.

Readings were taken on a stop watch as the needle of the galvanometer crossed each 0.05 of a millivolt on the galvanometer scale. These readings gave the total time as each division was

crossed by the needle. The same stop watch was used on all runs. The use of a magnifying glass gave accurate scale readings.

The current in the measuring circuit was checked against the standard cell at the beginning and end of each cooling and heating run. The temperature at the cold junctions was taken at the beginning and end of each run and, under ordinary room conditions, did not vary more than 1 degree Cent. This temperature was read on each of two mercurial thermometers, one attached to the vacuum cup at the cold junction and the other to a furnace leg about a foot away. Invariably the two thermometers indicated the same temperature.

Readings were started at 3 millivolts (400 degrees Cent.) (750 degrees Fahr.) and continued to 9 millivolts (980 degrees Cent.) (1795 degrees Fahr.) for the heating curve. The sample in the evacuated tube was allowed to reach a temperature between 1000 and 1010 degrees Cent. (1830-1850 degrees Fahr.) and then permitted to soak for 15 minutes. Readings for the cooling curve were started at about 8.5 millivolts (940 degrees Cent.) (1725 degrees Fahr.), except for very low carbon alloys which were started at 9 millivolts, and continued down to 5 millivolts (600 degrees Cent.) (1110 degrees Fahr.). Since no variations were observed upon heating through the range 400 to 600 degrees Cent. (750-1110 degrees Fahr.) it was considered unnecessary to continue to a lower temperature than 600 degrees Cent. (1110 degrees Fahr.) upon cooling.

The inverse rate method of plotting was used. The differences in time for each millivoltage interval were plotted against the total millivoltages. Cusps in the resulting curves indicated critical points. The millivoltage at the point of maximum deviation of the cusp was read, corrected, and the corresponding temperature recorded. The temperature-electromotive force table used was that supplied by the Bureau of Standards for the standard thermocouple. The heating curves are termed "C" curves and the cooling curves are termed "R" curves. All of the runs were made in a similar manner. The curves for sixteen of the thirty-two steels are presented in this paper and are grouped in order of the increasing carbon contents. All temperatures used are in the Centigrade scale. The rates of heating and cooling were the same—about 20 seconds per 0.05 millivolt. This amounts to 4 to 5 degrees Cent. in 20 seconds or 0.2

degrees Cent. per second. This rate was from two to four times slower than that employed in previous work on the iron-carbon diagram. Obviously the slower the rate of heating and cooling, the nearer the conditions are to equilibrium. (Figs. 10 to 13).

Reasons for the Choice of the Above Conditions: The following results from the work of previous investigators using thermal-analytical methods in connection with steels led to the choice of the conditions governing the runs made by the authors:

1. The same rate of cooling and heating must be used for all samples.

2. All specimens must be soaked at least fifteen minutes at the highest temperature before cooling.

3. A temperature of 1000 degrees Cent. (1830 degrees Fahr.) was most commonly used as a starting point for cooling.

Use of a higher initial temperature tends to lower and intensify the critical points. A temperature of 1000 degrees Cent. (1830 degrees Fahr.) must be used in low carbon steels to insure a constant rate of cooling by the time Ar_3 is reached. The same starting temperature must be used throughout in order to obtain the same rate of cooling for all samples and to maintain the same thermal conditions for each run.

4. A noble metal couple has been used with best results.

5. Cooling curves:

a. From a temperature just above the critical range these show a complete reversal of the changes on heating and are in keeping with thermal data.

b. In cooling from 1000 degrees Cent. (1830 degrees Fahr.) hypoeutectoid steels show little difference from the cooling curves started at temperatures just above Ac_3 for each steel. Above 0.80 per cent carbon a complete reversal had not always been apparent, but the A_{cm} point had been retarded and had occurred with a marked recalescence. When compared to curves started at lower initial temperatures the points were decidedly smaller.

6. The temperature from which the sample starts cooling and the time at temperature affect the critical points. Therefore these conditions have been kept constant for all runs for carbon contents under 1.20 per cent. For the two samples of 1.48 and 1.50 per cent carbon the starting temperature was raised to about 1060 degrees Cent. (1940 degrees Fahr.) in order to insure the normal

rate of cooling by the time Ar_{cm} for these two steels had been reached.

Working under these conditions it is interesting to compare the authors' results for carbonless iron with those of Burgess and Crowe[12] who found, as an average of 130 runs on their pure iron, that Ac_3 occurred at 909 ± 1 degree Cent. (1668 degrees Fahr.) and Ar_3

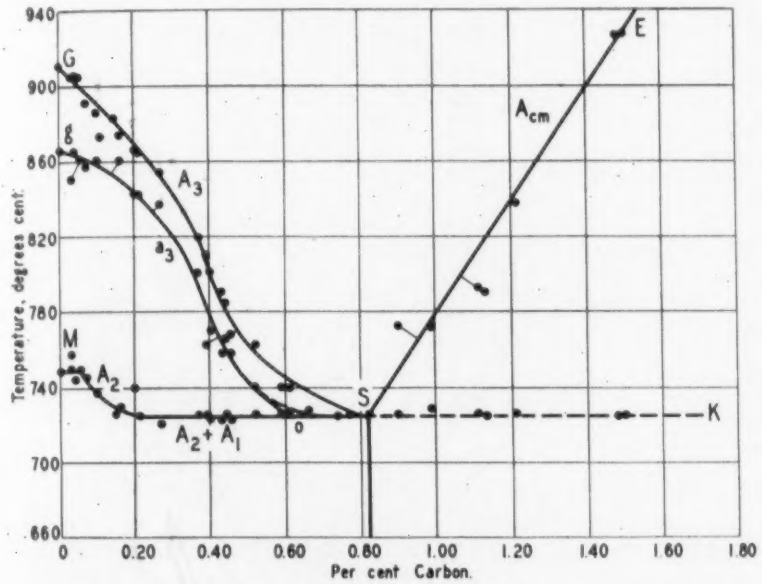


Fig. 6—Diagram Obtained by Plotting Points Observed on Heating the Specimens.

at 898 ± 2 degrees Cent. (1648 degrees Fahr.). Reading from the authors' curves for carbonless iron, Ac_3 was found at 910 degrees Cent. (1670 degrees Fahr.) and Ar_3 at 902 degrees Cent. (1656 degrees Fahr.).

7. Discussion of the Curves

The critical points from the "C" or heating curves are plotted against the carbon contents of the 32 steels in Fig. 6 which might be termed a "heating diagram."

The critical points from the "R," or cooling, curves are plotted against the carbon contents of the thirty-two steels in Fig. 7, forming a cooling diagram.

The thermal point diagram, Fig. 8, is an average of the points on the C and R diagrams.

[12] G. K. Burgess and J. J. Crowe, "Critical Ranges A_2 and A_3 of Pure Iron," Scientific Paper, No. 213, U. S. Bureau of Standards.

When the first heating curves were run on the carbonless iron sample it was noted that two cusps were obtained, one at 910 and one at 865 degrees Cent. (1670 and 1589 degrees Fahr.), the Ac_2 point having been observed at 749 degrees Cent. (1380 degrees Fahr.). Upon cooling, two cusps were found, one at 902 and one at 873 degrees Cent. (1656 and 1604 degrees Fahr.) before Ar_2 was

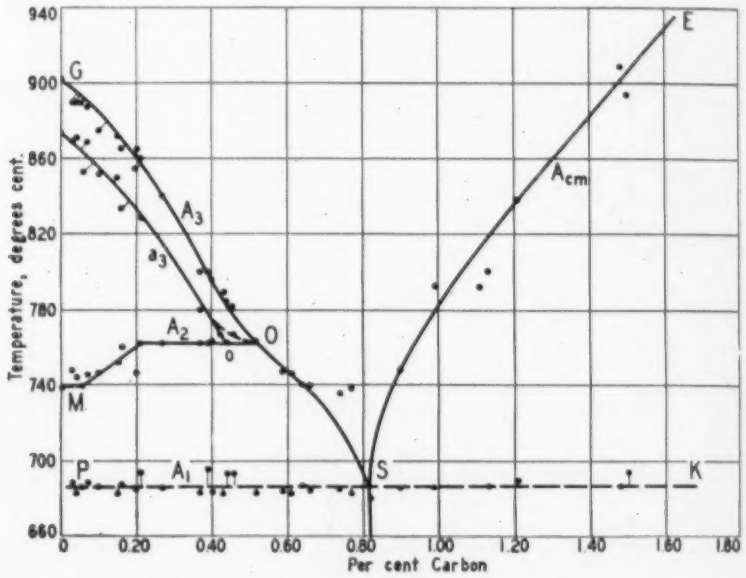


Fig. 7—Diagram Obtained by Plotting the Points Observed on Cooling the Specimens.

observed at 739 degrees Cent. (1362 degrees Fahr.). This double cusp was found in all the heating curves until the lower cusp merged with Ac_2 at 0.60 per cent carbon. The double cusp was found in all cooling curves until the lower cusp merged with Ar_2 for the 0.39 per cent carbon steel. On the thermal point diagram (Fig. 8) the average of the R and C lower cusps results in the line g-o merging with A_2 at about 0.53 per cent carbon. The average of the R and C upper cusps (which differed usually by only 3 to 5 degrees Cent. in lag) results in the line G-O in Fig. 8. In other words, the data seems to indicate that the A_3 change really takes place in two steps, the upper one being called A_3 along G-O and the lower one for convenience being called a_3 along g-o. It seems reasonable that more rapid rates of heating and cooling might result in the merging of the two into a single transition point.

In the carbonless iron sample Ac_2 occurred at 749 degrees

Cent. (1380 degrees Fahr.) while Ar_2 was observed at 739 degrees Cent. (1362 degrees Fahr.). With the carbon increasing to 0.07 per cent, these points occurred at about these same temperatures. However, from 0.07 to 0.15 per cent carbon, Ac_2 drops to about 722 degrees Cent. (1332 degrees Fahr.) where it apparently coincides with Ac_1. Ar_2 rises to about 762 degrees Cent. (1404 degrees

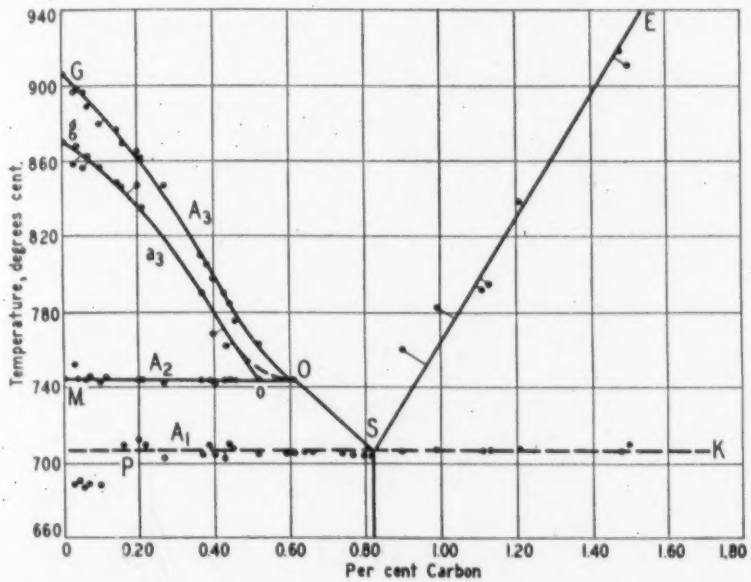


Fig. 8--Diagram Obtained by Averaging the Points on Figs. 6 and 7.

Fahr.) (the temperature observed on the cooling curves in previous researches) and remains constant at 762 degrees Cent. (1404 degrees Fahr.) until it meets Ar_3 (Fig. 7) at about 0.52 per cent carbon. Ac_2 remains combined with Ac_1 (Fig. 6) at 725 degrees Cent. (1337 degrees Fahr.) for carbon contents above 0.20 per cent. This variation may be due to an equal effect of some small amount of impurity on Ac_2 and Ar_2 for these average to give thermal point A_2 points that lie almost uniformly on the straight line M-O (Fig. 8).

The change corresponding to the A_1 change of the Carpenter and Keeling diagram (Fig. 1) is indicated as a dotted line P-K in Figs. 6, 7, and 8. This is done since it is not the authors' desire to discuss this transition at the present time. Further work is under way which it is hoped will elucidate the phenomena attendant upon the A_1 and $A_{3\text{-}2\text{-}1}$ changes. Results will be presented in a subsequent paper.

Beyond the eutectoid point, 0.82 per cent carbon, cusps are

observed on the heating and cooling curves at temperatures that become higher as the carbon content increases. These did not become consistent until a carbon content of 1.2 per cent was reached. The A_{cm} points on the curves for 0.99 and 1.13 per cent carbon are obviously inconsistent. Difficulty is usually met in determining A_{cm} close to the eutectoid. The data indicates that A_{cm} may justifiably be plotted as a straight line.

It will be noted that the 0.82 per cent carbon steel is apparently

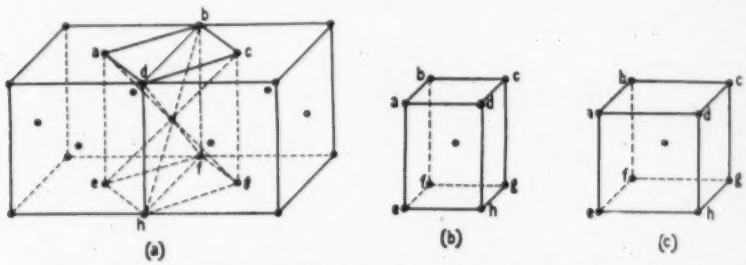


Fig. 9—Diagrams Illustrating Possible Atomic Rearrangements when Gamma Iron Changes to Alpha Iron or Vice Versa.

of eutectoid composition as the heating and cooling curves yield cusps that are concerned with only the A_1 change. Little variation from the data would result if the curve G-O-S were drawn as a straight line.

8. A Possible Two-Stage Transition at A_3

Some fundamental conceptions must first be considered:

a. Any finite rate of heating and cooling will only approach equilibrium conditions. Actually equilibrium may never be reached, —only to a practical degree.

b. Mobility of atoms and molecules, as such, in a solid phase is much less than in a liquid phase.

c. Allotropy—as it apparently exists in practically pure irons and steels—may involve two changes that are fundamental to other changes:

1. Some change in directional interatomic forces resulting from changes that may take place within the atom. (An inner-atomic change.)

2. The change of lattice dimensions dependent on the type of change in the directional interatomic forces. (An atomic space change.)

d. The heating and cooling curves of many steels and irons have shown *two* points at the commonly-termed A_3 point. The

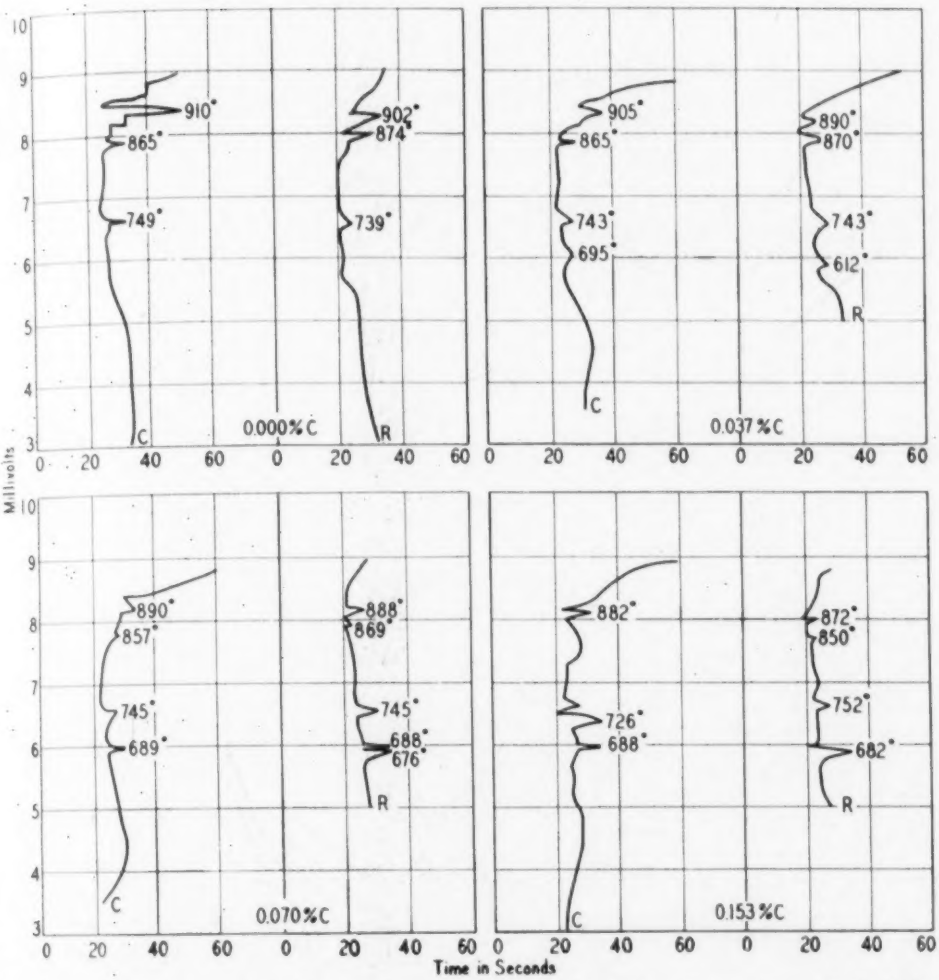


Fig. 10—Heating and Cooling Curves of Steels with Carbon Contents as Indicated.

series of steels used in the present research showed *two* cusps in every steel below the merging point with A_2.

e. Any finite rate of heating and cooling results in a certain "lag" effect. On cooling, a very small initial amount of the supercooled gamma iron changes directly to alpha iron at a temperature slightly below the true critical point. The amount of iron transformed and the temperature of its transition are largely determined by the rate of cooling—other factors being constant.

f. With an infinitely slow rate of cooling the transition points as shown by an inverse rate curve would disappear and only a slight deviation in the vertical curve might occur. This would be due to the averaging of specific heats of alpha and gamma irons

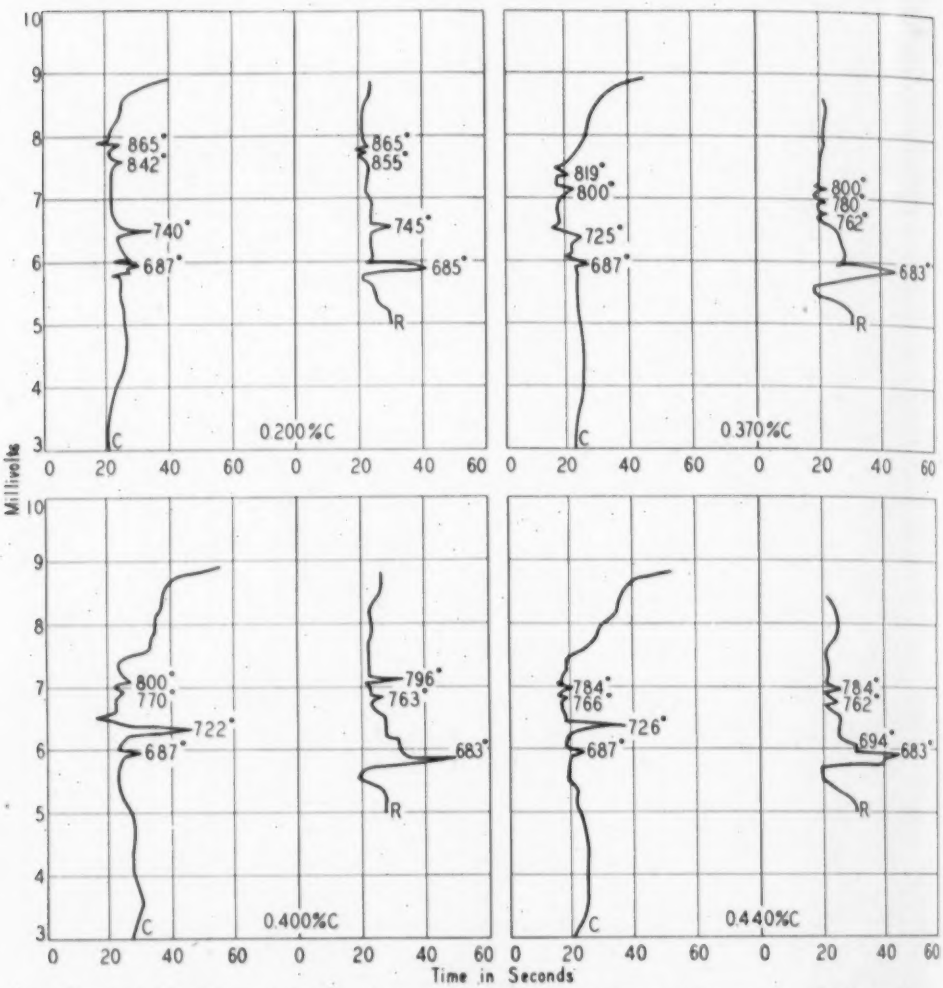


Fig. 11—Heating and Cooling Curves of Steels with Carbon Contents as Indicated.

as one changes to the other. This deviation would probably be so small as to elude observation by present methods of analysis.

g. In a solid phase with a finite rate of cooling, might not the mobility of the units in the solid phase be such that the lattice dimensional changes might lag a bit behind the directional changes of the interatomic forces? Thus two separate energy transformations might result in two transition points on the cooling and heating curves. Is this not a working hypothesis for the explanation of the occurrence of a two-cusp A_3 change?

h. Thus on heating and cooling diagrams the double line should be drawn for A_3. It is conceivable that under absolute equilibrium conditions of infinite time at temperature the two-cusp

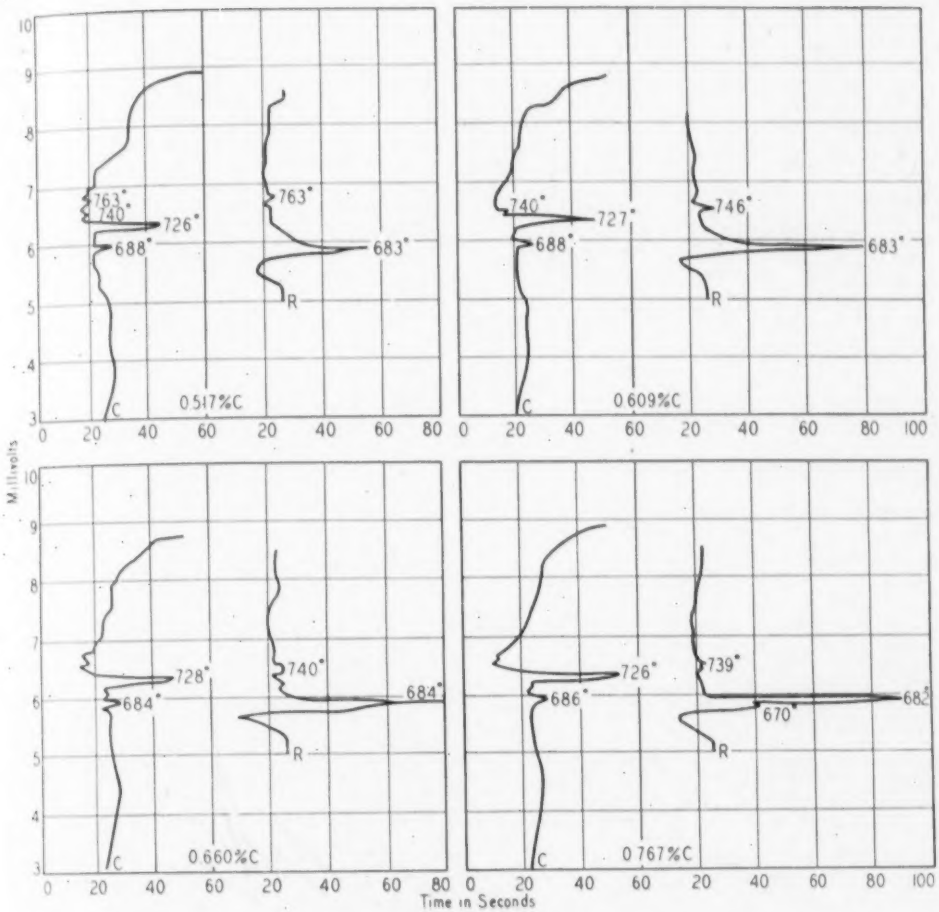


Fig. 12—Heating and Cooling Curves of Steels with Carbon Contents as Indicated.

change would merge to a single critical equilibrium point,—the lattice change being coincident with the interatomic force—direction change. However, for the present, a double line is presented on the averaged, or thermal point, diagram in order that the data be unchanged by interpretation.

Thus, a possible application of this interpretation as presented by the authors would be as follows:

It is generally accepted that Ac_3 represents the end of the reabsorption of alpha ferrite into austenite and Ar_3 the beginning of the precipitation of alpha ferrite from austenite. In the present work, as already indicated, the A_3 change was constantly observed to take place in two steps as shown by the double cusp on the curves. Check runs as well as original runs confirmed this observation. In the succeeding paragraphs the authors have attempted an illustra-

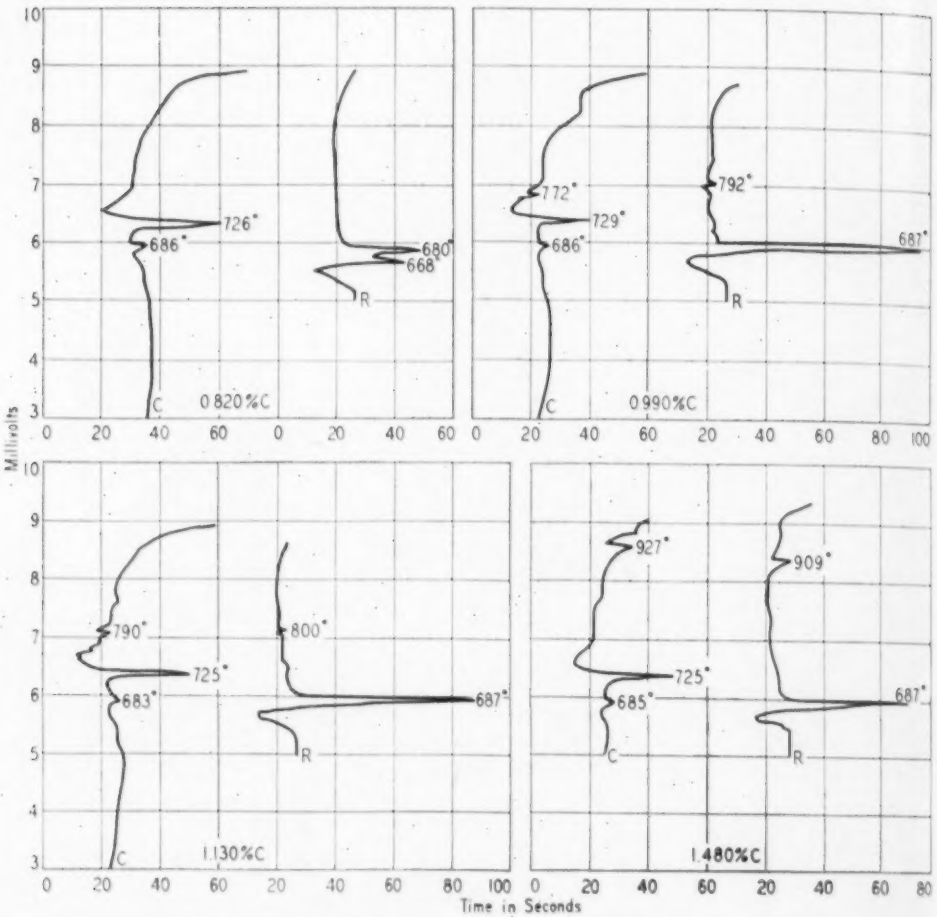


Fig. 13—Heating and Cooling Curves of Steels with Carbon Contents as Indicated.

tion of an application of this hypothesis in terms of the atomic changes during the allotropic change. The diagrams in Fig. 9 were obtained from a paper entitled "The Nature of Martensite" by E. C. Bain.[13]

X-ray studies have shown gamma iron to possess a face-centered cubic structure with a side length, in the elementary cube, of 3.60 Å. Alpha iron has a body-centered cubic structure with a side length in the elementary cube of 2.86Å. Thus when gamma iron changes to alpha iron two changes are involved: (a) the reference positions in the elementary cube change from a face-centered linkage to a body-centered linkage, and (b) the size of the unit cube decreases.

Fig. 9 is a diagrammatical representation of what might take place. Diagram A in Fig. 9 indicates how the atoms might be re-

[13]Edgar C. Bain, "The Nature of Martensite," *Transactions* American Institute of Mining and Metallurgical Engineers, Vol. LXX, 1924, p. 25.

arranged as the face-centered system changes to the body-centered system during the Ar_3 change. A body-centered unit a-b-c-d-e-f-g-h may be visualized including a common face d-b-f-h of two adjacent face-centered cubes. The body-centered unit a-b-c-d-e-f-g-h shown in diagram B in Fig. 9 has momentarily a side length a-b equal to one-half the diagonal of the gamma cube, or 2.55Å, and an altitude d-h equal to the side of the gamma cube which is 3.60Å. This might constitute the first step in the change as denoted by the upper cusp in the cooling curves. The system has the body-centered structure but is not yet cubic and on the thermal point diagram this change is noted as A_3.

The alpha iron cube is shown in diagram C in Fig. 9. The second or lower step of the change from gamma to alpha iron might consist of the lengthening of a-b and shortening of d-h until both have a length of 2.86Å. This change might account for the a_3 line as shown on the cooling diagram.

It is quite important to remember that a trace of some element too small in quantity to be discovered by analysis—or eluding analysis—may be responsible for separating the A_3 change into two cusps observed and interpreted. Thus in a series of steels of even greater purity these two energy changes might merge to a single transition point. An increase in some other impurity might have the effect of causing mergence of the two steps.

9. Summary and Conclusions

1. A series of thirty-two very pure iron-carbon alloys has been assembled and thermal analyses made of them in a modified Rosenhain furnace. The carbon contents of these alloys vary from 0.00 to 1.5 per cent. This series constitutes one of the most complete sets of carbon steels used in attempting to locate the transition points in the steel area of the iron-carbon diagram.

2. Heating and cooling diagrams has been plotted from the curves obtained in the thermal analyses. The inverse rate method of plotting has been used. A thermal point diagram has been plotted by averaging the associated points on the heating and cooling diagrams.

3. Evidence was obtained that the A_3 change may take place in two steps. The lower step for convenience has been called the a_3 change. An explanation for this possible two step transition has been offered.

4. The merging point of A_3 with A_2 occurs at about 0.60 per cent carbon rather than at 0.38 per cent carbon as is shown on most diagrams.

5. The steel containing 0.82 per cent carbon gave the best evidence of being the eutectoid alloy.

6. The slopes of the A_3 and $A_{3\text{-}2}$ ranges seem to be about the same, which justifies drawing them in as a straight line rather than a broken line.

7. Points located in the A_{cm} range furnish as much justification for drawing it in as a straight line as a line having varying slope.

8. A_2 under the given conditions appears to be located about 20 degrees Cent. lower than on published diagrams based only on cooling curves.

9. No attempt was made to study the occurrence of the so-called A_0 range at 215 degrees Cent. (419 degrees Fahr.).

10. While considerable data were assembled concerning the A_1 and $A_{3\text{-}2\text{-}1}$ ranges, the authors felt that further study must be made inasmuch as these transitions involve more complicated phenomena than heretofore reported.

DISCUSSION

O. W. Ellis:[1] I was reminded, in reading this paper, of considerable discussion which I have read, but did not hear, which had taken place in the meetings of the British Iron and Steel Institute. It is not impossible that Dr. Waterhouse himself has heard these discussions or read them. I cannot quote exactly the year in which these discussions took place, but I do know that for quite a considerable time Professor Arnold of Sheffield was convinced that the A_2 transformation was double in its nature; in other words, that 2 cusps occurred at the A_2 point in heating and cooling curves of iron. So far, however, I have seen no satisfactory explanation of Professor Arnold's results, though his views regarding the duplex character of the A_2 point were generally discounted. On the fifteenth page of this paper, the authors state that it seems reasonable that small amounts of impurities and more rapid rates of heating and cooling might result in the merging of the two into a single transition point. I would like to suggest that they may have overlooked the effects of specimen shape or size on the heating and cooling curves which they have obtained. I know that considerable differences in the appearance of cooling curves and heating curves can be obtained as a result of alterations in the shape and size of the specimens being used. I know that double cusps can be produced with certain shape specimens and that single cusps can be produced with other shape speci-

[1]Director of metallurgical research, Ontario Research Foundation, Toronto, Ont., Canada.

mens. I would suggest that the authors investigate this possible cause of the double cusp before deciding definitely that the A_3 point does occur in the manner in which they have suggested. I am not, of course, putting forth the suggestion that their discoveries are not correct, but I am suggesting that it might be well for them to investigate the above possibility, which apparently has not been looked into, in view of their statement at the bottom of the fifteenth page.

There is one thing which I am very much surprised at, and that is the position of the A_2 point. I have run heating and cooling curves on innumerable samples of low carbon steel, but I have never been able to get an A_2 point as low as 740 degrees Cent., and none of those with whom I have been associated in the various places where heating and cooling curves have been taken have ever found as low a temperature as that—740 degrees Cent.—for the A_2 point.

R. S. ARCHER:[2] The importance of this subject is certainly obvious to all of us. It seems unfortunate, and in fact surprising, that there has been so little work done on the constitution of the pure iron-carbon alloys since the comparatively early work referred to by the authors. We must, therefore, welcome any attempt such as this to further establish the lines of the constitution diagram. Perhaps the first consideration is that the authors have put before us certain facts. Regardless of interpretation, they have presented the results of numerous cooling and heating curves on a series of steels which is somewhat unusual as to manner of preparation. The steels have the advantage of having been prepared from a similar, or practically identical, base material by a process which does not involve remelting.

Perhaps the most conspicuous feature of these results is the double cusp found for the A_3 point. Dr. Ellis has already called attention to the possibility that this observation might have been caused by an unsuitable form or size of sample. The authors have advanced an explanation for this double cusp, assuming, of course, that it really occurs. In addition to the point mentioned by Dr. Ellis, it may be well to keep in mind two of the possible errors of the Rosenhain method of taking thermal curves. These errors have both been eliminated in certain work with this type of furnace, but I believe many of us have seen other examples of work in which the errors have not been eliminated. The two errors to which I refer are (1) discontinuities in the rates of heating and cooling due to mechanical causes, that is to a discontinuous upward or downward motion of the specimen in the furnace; and (2) the possibility of parasite currents in the thermocouples. In this method a thermocouple is used which has a constantly changing depth of immersion, and as we all know it is easier to avoid thermocouple difficulties by maintaining a constant immersion depth.

The authors' explanation of this splitting of A_3 is that the energy change is discontinuous. Specifically, they attribute an energy change to what they call the first step of the allotropic transformation as illustrated in the diagram taken from Mr. Bain's suggestions. One's first impression of that diagram, I would think, is that what the authors have regarded as the first step in the transformation is nothing but a different way of looking at the iron crystal. We take a face-centered cubic crystal and show that by selecting certain atoms

[2]Metallurgical laboratory, Aluminum Company of America, Cleveland.

and looking at them in the right direction we have a structure from which the body-centered cube can be derived. It would seem that what they called the first step of the transformation is merely a change in our mental aspect toward the gamma iron structure. Perhaps they really mean that this is not merely a changed aspect but that the first thing that happens is a change within the iron atoms which causes a shifting in what they have referred to as the direction of the forces between the atoms. That leads to the question of the nature of this supposed change within the iron atoms. It has been suggested that such a change is responsible for the A_2 transformation. I believe that in most respects A_2 is a gradual change rather than a sharp change, and that the point observed on heating and cooling curves represents a maximum rate of change rather than a definite temperature above which we have one kind of atom and below which we have another. The changes in magnetism are such as to indicate this. Pure iron does not lose its magnetism abruptly at A_2, but there is a fairly rapid decrease in permeability well below the A_2 temperature, the rate of change reaching a maximum at that temperature. If this is the case, we might expect that the atoms have changed entirely, or nearly entirely, to the form favoring the face-centered cubic arrangement before the actual A_3 point is reached on heating. This seems to constitute another difficulty.

The authors have also suggested that this doubling of the point may be caused by impurities. It would seem that this is the most probable explanation, if we assume that the experimental methods were such that the reported doubling represents actual changes in the steels. In a paper presented to the Society within the last year or two by Hultgren, there is an excellent demonstration and discussion of what we might call the doubling of the A_1 point. As a matter of fact, Hultgren shows that A_1 can take place over a wide range in temperature. He shows rather conclusively that this can frequently be attributed very largely to segregation or a lack of homogeneity within the steel. The effect of the size of the bar from which the specimen was cut was significant. On a bar about 3½ inches in diameter he found a wide A_1 range on a specimen from the center of the bar and a narrow range on a specimen from the outside. When similar steel was rolled to a smaller size, the A_1 temperature range was narrow all through the specimens, indicating that lack of homogeneity had been removed by the mechanical and thermal treatment.

Supposing impurities to be important, it becomes of interest to question the impurities that may have been present in these steels. I hope that someone from the American Rolling Mills Company can give us some definite information on the impurities apt to be present in this material. I believe that the samples included Armco iron of different dates of manufacture, some of it going back about six years and some of more recent manufacture. In particular it would be of interest to know the probable copper content and the probable oxygen content. Dr. Grossmann has shown us this morning the very interesting and important effects that oxygen may have in steel. In this connection it may be quite worth while to consider the possible effects of the hydrogen used in producing these steels. These steels were all made by annealing in hydrogen, and it seems quite likely that there may have been significant changes in oxygen content during that annealing.

Now as to other evidence on the points in steel, and particularly pure

iron. As Dr. Ellis has pointed out, A_2 is usually found at a higher temperature than the authors have found. It would seem very interesting if some of the men from the Bureau of Standards could tell us what they have found. They have run a great many heating and cooling curves, and a good many of them on electrolytic iron. I believe Burgess and Crowe report a temperature of 768 degrees for A_2 and they found very little difference between the temperature on heating and cooling.

The authors have averaged their points on heating and cooling and as a general proposition it is considered that the temperature obtained on heating is closer to the equilibrium temperature than that obtained on cooling.

It would be most satisfactory if the authors themselves could check some of this work with samples of electrolytic iron, and perhaps with a method of heating and cooling in which the specimen is stationary instead of moving as in the Rosenhain furnace. In spite of these points, however, I think we should be very grateful to the authors for the considerable amount of work they have done on an important subject.

Authors' Closure

The authors wish to express their sincere appreciation of the discussions brought forward by Dr. O. W. Ellis and Mr. R. S. Archer.

First, in answer to Dr. Ellis: although the A_3 and A_2 transformations fall in the same class—that of inner-atomic changes—the A_3 point is further characterized by a shifting of the lattice. For this reason, it is difficult to see just what direct bearing Professor Arnold's duplex views on the A_2 change may have on the A_3 change in the light of the present paper.

The statement at the bottom of the fifteenth page and correctly quoted by Dr. Ellis has a meaning unaffected by any consideration of the shape of sample used. However, Dr. Ellis is undoubtedly right in pointing out that the shape of the specimen may affect the thermal curves. The authors used samples of two shapes: (a) $\frac{5}{8}$ inches long by a circular $\frac{1}{4}$ inch cross section and (b) $\frac{5}{8}$ inches long by a square $\frac{1}{4}$ inch cross section. Both shapes yielded the 2-cusp A_3 within certain carbon percentages. If the double cusp A_3 is the result of the specimen shape, why is the double cusp lacking on the cooling curves for steels between 0.53 and 0.82 per cent carbon contents?

The position of the A_{cr_2} points—as is mentioned in the paper, and also pointed out by Mr. Archer—"may be due to an equal effect of some small amount of impurity for these points average to give thermal point A_2 points that lie uniformly on the straight line M-O (Fig. 8)."

Second, in reply to Mr. Archer: the authors appreciate that the Rosenhain method may be subject to the two errors of mechanical discontinuities and parasite currents in the thermocouple. The operator of a machine, after making many runs, develops either a sense of confidence substantiated by consistent results, or a feeling of doubt caused by erratic results. In blank runs there were no variations in the curves that approached in degree and character even that of the smallest A_3 point. It would seem strange that mechanical errors or couple parasite currents could cause A_3 to occur as a double cusp with such consistent variation with the carbon content.

It is perhaps unfortunate that Mr. Bain's diagrams were used to illus-

trate one possible interpretation of the author's double cusp A_3 transformation. The situation will undoubtedly be clarified if the lines connecting the atom positions in these diagrams be considered as possible force lines. Thus, on cooling, a change within the iron atom causes a directional change in the force lines emanating from the atom and this, in turn, necessitates a shifting in the lattice toward a new state of equilibrium. Thus a 2 cusp A_{r3} may, under some conditions, arise from a lag of the lattice shift behind the inner atomic change. Whether the two possible energy changes be separated by certain heating conditions or by the effect of some impurity present in small amount, all of the evidence seems to point to the double cusp as being an A_3 effect, varying consistently with the carbon content and merging into a single cusp after junction with A_2.

As Mr. Archer states, Hultgren did find a lack of homogeneity in a bar of steel 3¼ inches in diameter. The samples used in the authors' work were prepared in ¼ inch cross section stock and the method of preparation required from 6 to 10 days at 960 degrees Cent. followed by cooling in the furnace. Microscopic inspection showed the carbon distribution to appear uniform.

These long heats at 960 degrees Cent. took place in hydrogen which should have acted at least partially to eliminate the oxygen. In the discussion of Dr. Grossmann's article on "Oxygen Dissolved in Steel", TRANSACTIONS, American Society for Steel Treating, July, 1929, are thermal curves for very low carbon alloys with iron. These curves were run by H. Scott, Miss I. J. Wymore, and H. C. Cross on Arsem melted electrolytic iron plus about 0.05 per cent carbon, on ladles of these alloys treated with aluminum to eliminate oxygen as far as possible, and on ladles untreated with aluminum. From one to four of the curves obtained by each investigator showed double cusp A_3 points. It would seem that oxygen is not involved in this phenomenon.

We cannot say definitely that hydrogen may not be involved. Yet it seems that check runs on samples already heated for a time in vacuum at 1000 degrees Cent. ought to be free from any effect of hydrogen.

The averaging of corresponding A_3 points on the heating and cooling diagrams seems entirely permissible since the lag between the A_r and A_c points is usually within the error of the thermocouple, ±3 degrees Cent., or a range of 6 degrees Cent.

Finally, the evidence, at present, seems to lead, as one of the conclusions, to the possible occurrence of A_3 as a two-stage transformation. If A_3, under some conditions, occurs in two steps, this might have some bearing on the formation of the different types of martensite recognized by Hanemann and Schrader in their paper "On Martensite", TRANSACTIONS, American Society for Steel Treating, Vol. 9, p. 169. If two steps be separated, even only slightly, might this not (together with interstitial carbon atoms) cause warping of the body-centered lattice of martensite resulting in the diffuseness of X-ray patterns from martensite? Heindlhofer and Bain in their article on "The Grain Structure of Martensite", TRANSACTIONS, American Society for Steel Treating, Vol. 18, p. 106, describe alpha iron in hardened steel as possessing "some characteristic imperfections, distortion, or crystalline idiosyncrasy" as departing "from cubic symmetry represented by a tetragonal system". Many other interesting possibilities would take up too much time and space at this time.

METASTABLE EQUILIBRIUM IN HYPEREUTECTOID IRON-CARBON ALLOYS

By A. Allan Bates, D. E. Lawson and H. A. Schwartz

Abstract

The paper constitutes a continuation of certain work reported in "Graphitization of Prequenched White Cast Iron," Schwartz, Johnson and Junge.

The composition of the two phases in equilibrium in the metastable system between the eutectoid and eutectic temperatures was determined by a planimetric study of two binary alloys differing in carbon content. It is shown that while the location of the A_{cm} line is confirmed by this study, the composition of the carbide phase does not agree with the formula, Fe_3C, above the eutectoid point. The explanation is believed to be a solution of gamma iron in the carbide phase.

Preliminary observations on the possible solubility of carbon in iron carbide are also noted.

THE object of this investigation is twofold: (1) The hypothesis has recently been advanced that the iron carbide (cementite), precipitated during cooling of hypereutectoid iron-carbon alloys between the eutectic and eutectoid temperatures, is not a definite compound of the formula Fe_3C, but is of varying composition. One of the objects of this investigation is to collect data bearing on the substantiation or refutation of this hypothesis. (2) The exact location of the A_{cm} line of the iron-carbon equilibrium diagram is at present a matter of some uncertainty. It is a secondary object to determine the location of this line by a new method.

Schwartz et al[1] described a method for the calculation of the carbide content of the two phases of iron-carbon alloys in equilibrium at a given temperature and suggested a decrease in carbon content of the carbide phase with rising temperature in the presence of some 0.35 per cent silicon.

[1]H. A. Schwartz et al; "Graphitization of Prequenched White Cast Iron," presented before the A. S. S. T. in Cleveland, September, 1929.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The authors are members of the society. The experimental work here recorded was undertaken as an activity of Dept. of Metallurgy, Case School of Applied Science, in partial fulfillment of the requirements for the degrees of M. S. and B. S. in Metallurgical Engineering by Bates and Lawson respectively.

It has been deemed advisable to apply the new method of investigation to essentially pure iron-carbon alloys free from silicon, manganese and the other common impurities found in white irons. To this end the present investigators have used materials derived from the alloying of washed metal and Armco ingot iron which, by chemical analysis, have been found to be essentially free from the above mentioned impurities.

Preparation of Alloys

The melts were made in special magnesite-lined graphite crucible. Such a crucible charged with wash metal or wash metal and Armco iron, was charged into the Arsem furnace. After the vacuum pump had reduced the pressure in the furnace to about one-eighth of an inch of mercury, the current was started at about 90 amperes. The charge was left heating at this rate for about ten minutes in order that the magnesite lining of the crucible should not be cracked by too rapid a rise in temperature. The current was then increased at the rate of ten amperes every five minutes until 150 amperes were reached. At the resulting temperature (about 3000 degrees Fahr.) the alloy was in the molten state, and due to the high temperature, combined with the low pressure, the metal boiled and in this way was freed from occluded gases. The metal in every case was solidified and cooled to blackness in the furnace under vacuum, thus assuring no oxidation.

Heat Treatment of Specimens

The specimens finally used for heat treating were in the form of cylinders about ½ inch long and ½ inch in diameter. These were placed in the Rosenhain quenching furnace in pairs, a low carbon and a high carbon specimen in each pair. The vacuum pump was attached to the apparatus and allowed to run until a sufficient vacuum was reached to cause a small amount of water placed in a suction flask to boil. The current was then turned on and increased at such a rate that approximately two hours' time were required to arrive at the desired temperature. After this point was reached, a twenty minute period was devoted to soaking the specimen at a constant temperature. It was possible to hold the furnace within a range of nine degrees Fahr. At the end of the soaking period, the specimens were quenched and removed from the suction

Table I
Carbon Analyses

Heat No.	4	C 3.48%
Heat No.	5	C 2.94%
Heat No.	7	C 2.84%
Heat No.	13	C 2.34%
Heat No.	19	C 1.72%
Heat No.	20	C 1.62%

flask. All surfaces of the specimens were ground down from two-hundredths to five-hundredths of an inch in order to assure removal of all oxide and any decarburized shell which might have been formed.

Chemical Analyses

In Table I there has been included the carbon analysis of only those Arsem furnace heats which were used in the final determinations of points on the iron-carbon equilibrium diagram. These determinations were made by the combustion method.

Each of the heats in Table I gave an ingot which, by microscopical examination, showed no chemical segregation. Had such segregation been present, planimetric determinations would have given varied results in accordance with the face of the specimen chosen for photomicrography. In order to obtain further assurance of the absence of segregation, one of the ingots, No. 7, was analyzed at both top and bottom. The results gave excellent corroboration of those arrived at by metallographic examination.

As previously stated, the raw materials for the Arsem furnace were washed metal, and in some cases, small proportions of Armco ingot iron. Analyses run previously to the present investigation showed that alloys made of these materials by the method chosen for this research would fall within the following limits with regard to impurities:

Silicon	less than 0.03 per cent
Phosphorus	less than 0.03 per cent
Sulphur	less than 0.03 per cent
Manganese	less than 0.01 per cent

Close examination of many of the Arsem furnace ingots convinced the authors that these ingots were exceptionally sound and free from blow-holes. However, in order to prove such soundness, several of the specimens were subjected to X-Ray examination[2]. No

[2]By Dr. Kent Van Horn.

Table II
Observed Values

Heat Treatment No.	Arsem Furnace No.	Temp. of Heat Treat. Degrees C.	Density	Traverse Carbide Phase	Traverse Solution Phase
1	19	As cast	7.716	24.53	135.47
2	19	768	7.835	31.15	138.85
3	19	816	7.666	19.56	140.44
4	19	865	7.700	19.43	140.57
5	19	914	7.663	9.39	150.61
6	19	962	7.670	6.22	153.78
7	19	1010	7.631	.....	.....
8	19	1060	7.520	4.62	155.38
9	13	As cast	7.733	20.63	59.37
10	5	768	7.682	60.22	99.78
11	5	816	7.541	52.96	107.04
12	5	865	7.557	52.25	107.75
13	5	914	7.482	37.56	122.44
14	5	962	7.455	42.13	117.87
15	5	1010	7.439	.....	.....
16	5	1060	7.512	.....	.....
17	4	914	7.735	71.17	88.83
18	4	914	7.723	67.40	92.60
19	4	914	7.708	77.05	82.95
20	7	914	7.547	51.45	108.55
21	20	914	7.780	13.66	146.34
22	19	914	7.808	11.02	148.98
23	5	As cast	7.577	45.41	74.59
24	7	1060	7.461	48.05	111.95

blow-holes or cavities, however, are indicated in any specimen. In Table II will be found the results of density determinations and of intercept planimeter measurements on the several samples. In Table III are shown the corresponding carbon concentrations derived by calculation from Table II. For the method of calculation the first reference may be consulted.

Calculation of Results by Method of Least Squares

Using the method of least squares, the positions of the A_{cm} line and of the "carbide" line were calculated from the data collected during the present investigation.

Letting C = per cent of carbon
and T = temperature in degrees Cent.

the equation of the A_{cm} line becomes (using all values of Table II)

$$C = -.652 + .00216\ T \qquad (1)$$

and the equation of the "carbide" line becomes (excluding data involving melt No. 5, or temperatures above 914 degrees Cent.)

$$C = 7.834 - .00182\ T \qquad (2)$$

Substituting for values of T in the above equation, we find that at 700 degrees Cent. (1290 degrees Fahr.) the carbide concentration is equal to 6.57 per cent of carbon, while for the A_{cm} line it is equal to 0.86 per cent. At 900 degrees Cent. (1650 degrees Fahr.) the

Table III
Calculated Values

Heat Treatment No.	Temp. of Treatment	Carbon Concentration Solution Phase	Carbon Concentration Carbide Phase
1 vs. 9	As cast	.806	6.66
1 vs. 23	As cast	.914	6.56
2 vs. 10	768	1.08	6.29
3 vs. 11	816	1.03	7.14
4 vs. 12	865	1.02	7.24
5 vs. 13	914	1.33	9.01
6 vs. 14	962	1.45	7.43
8 vs. 24	1060	1.61	6.73
5 vs. 17	914	1.26	6.18
22 vs. 19	914	1.43	5.76
21 vs. 19	914	1.15	6.81
5 vs. 20	914	1.47	5.92
5 vs. 18	914	1.43	6.23

carbon concentration on the A_{cm} line is 1.29 per cent, and on the carbide line, 6.19 per cent. Assuming both lines to be straight, and extrapolating them to 1130 degrees Cent. (2065 degrees Fahr.) the eutectic temperature, we find the carbon concentration on the A_{cm} line to be 1.79 per cent, and on the carbide line, 5.79 per cent.

If, instead of postulating a straight A_{cm} line as above, we assume that the solubility C (in %) is related to the absolute temperature, T_a, according to Tammann's equation

$$\log C = a/T_a + b$$

we may evaluate a and b by the method of least squares and derive the equation

$$\log_{10} C = 0.987 - 1021.49 \frac{1}{T_a} \qquad (3)$$

No similar equation for the solubility of gamma iron in cementite is possible for at A_1 apparently there is no solubility, i.e. $C = O$ hence $\log C = -\infty$ which cannot be true for finite values of a and b in the Tammann equation.*

It is probably hopeless to distinguish experimentally between the validity of (1) and (3) respectively for the locus of (3) is so nearly a straight line as to be indistinguishable therefrom within the limits of error of the values of C.

Discussion and Conclusions

The results of the investigation seem to point unmistakably to one fact, namely, "cementite" does change in composition as it is

*It is recognized that the Tammann equation refers only to molecular concentrations. In the absence of knowledge as to the molecular weight of the substances entering into austenite, Equation 3 has value only to the extent that since C is not very large, the molecular concentration of carbon will be reasonably nearly in proportion to its concentration by weight.

heated to progressively higher temperatures above the A_{3-2-1} line. The nature of this change has not been definitely shown by the present investigation. As a matter of fact, the data collected seem to indicate that the nature of the change may be more complex than was at first supposed, and may vary in accordance with variations of certain conditions not as yet defined. The results of our calculations bring us to several rather definite and very important conclu-

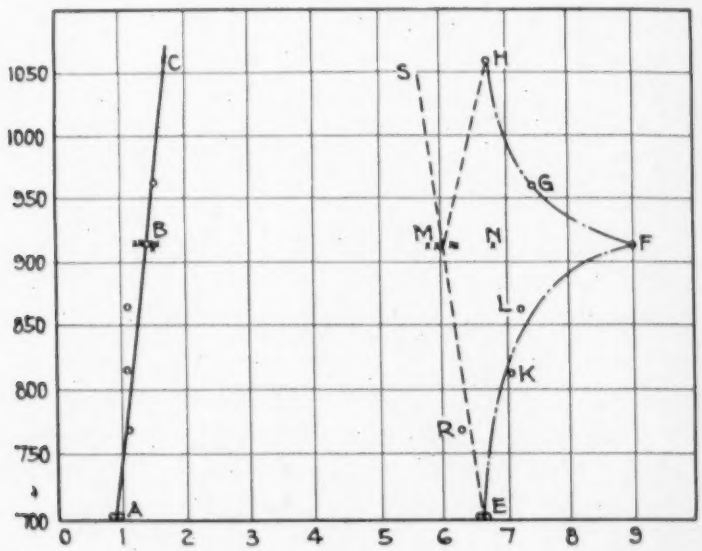


Fig. 1—Graphic representation of the results of the investigation, showing positions of A_{cm} line and of "carbide" line is calculated by the method of least squares.

sions. In addition, they suggest a number of interesting speculations which, it is hoped, will be valuable in pointing out paths of procedure for future additional research.

Turning now to the diagram, Fig. 1, some explanation and discussion of the curves shown are necessary before proceeding to conclusions. The points represented by dots within circles were obtained by successive heat treatments carried out on the same pair of specimens (Nos. 5 and 19). The "as cast" specimens were planimetered, subjected to density determinations, and the results calculated and plotted at a temperature corresponding to the A_{3-2-1} line. The resulting points, together with corroborative investigations carried out on specimens 19 and 13 "as cast," show a remarkable agreement with the commonly accepted values of the carbon concentrations of the phases existing at the A_{3-2-1} line. Thus, the average of the values obtained for the eutectoid point is 0.86 per cent carbon,

while the average of the values for "cementite" is 6.61 per cent.

Here we may draw our first conclusion: the new method of research is capable of giving accurate and trustworthy results. If further proof of this is necessary, it may be pointed out that the median line of all the points determined along the A_{cm} line is in the same location as the commonly accepted location of that line. Extrapolated to the eutectic temperature of 1130 degrees Cent. (2065 degrees Fahr.), our results show that the carbon concentration of saturated austenite at that temperature, is equal to 1.79 per cent.

The treatments of specimens 19 and 5 at a temperature of 768 degrees Cent. (1415 degrees Fahr.) showed the carbide to be tending to decrease in carbon concentration. Subsequent treatments, at progressively higher temperatures, showed a remarkable increase in the carbon concentration of the carbide phase, culminating in value of 9.01 per cent, at a temperature of 914 degrees Cent. (1675 degrees Fahr.). It may be noted in passing that this approaches closely the percentage of carbon in Fe_3C. Now let it be recalled that the curved line EF is derived from simultaneous equations involving values of points lying along the line ABC. It is very unlikely that the extraordinary values obtained for the points along the line EF could have arisen from an error since that would almost certainly have involved a compensating error, obviously not present, in the values obtained for points along the line ABC.

The treatments carried out on the specimens 19 and 5 at 962 degrees Cent. (1765 degrees Fahr.) showed the carbide phase returning to lower carbon concentrations. This surprised the investigators until, on examination at high magnification, the high carbon specimen showed signs of graphitization. Subsequent treatments at 1010 and 1060 degrees Cent. (1850 and 1940 degrees Fahr.) exhibited unmistakable progressive graphitization. The particles of graphite formed, were extremely fine in size, a magnification of 1000 diameters being required to resolve them. This matter of graphitization will be recalled to the reader's attention later in the chapter.

It was obviously useless to proceed with the planimetry upon the specimens which showed an appreciable amount of graphitization, but it was thought highly desirable, if possible, to obtain a point near the eutectic temperature. For this purpose a new high-carbon specimen, No. 7, was treated in conjunction with No. 19 at 1060 degrees Cent. (1940 degrees Fahr.). The resulting points with

values of 1.61 per cent carbon for the austenite phase, and 6.73 per cent carbon for the carbide phase, again show continuity when considered in conjunction with the previously determined points.

Since the point F showed the maximum inflection of the curve, it was next thought desirable to check the treatment at 914 degrees Cent. (1675 degrees Fahr.). A new high-carbon specimen, No. 4, not previously heat treated, was run at this temperature. The resulting point in the carbide line, showed the curve to bend in the opposite direction to the point F. The question now arose, had treatments K and L in Fig. 1 been instrumental in causing the carbide line to swing out to the point F, and further, would repeated treatments of specimen No. 4 at 914 degrees Cent. (1675 degrees Fahr.), cause it likewise to show a phase with about 9 per cent of carbon? In other words, does time play an important part in the location of the carbide line?

In order to answer this question, treatments were repeated on specimen No. 4, the third treatment involving a hold of five hours at this temperature. After each treatment, calculations were made so that, if possible, the effect of the time factor could be quantitatively observed. The carbide line, however, showed no tendency to repeat its original performance of swinging out to the point F. Further checks were made by repeating the 914 degrees Cent. (1675 degrees Fahr.) treatment on two new specimens, one of low carbon, No. 20, and one of high carbon, No. 7. These were calculated against each other, and were also cross-calculated with the specimens previously used. The resulting points, with one exception, N, grouped themselves closely about a value of 6 per cent for the carbon concentration of the carbide phase. The corresponding values on the A_{cm} line likewise checked closely, with on exception, this point corresponding with N. Because of this double error, the point N has not been taken into consideration in the location of the line EM. However, in order that the authors may not be accused of choosing points to suit their convenience, it may be noted that, even taking point N into consideration, the average value of the carbon concentration of the group at M is changed but slightly from the value of 6 per cent.

It is worthy of remark that the point R, which is the only point of the group RKLFG, which had not received one or more previous heat treatments, lies rather close to the line EM. The authors be-

lieve that it is a point of particular importance to note that none of the specimens along ERMH showed graphitization, whereas unmistakable graphitization showed at G and at subsequent heat treatments on the same specimens as those upon which G was calculated.

We are now in position to proceed with definite, though perhaps tentative, conclusions. First, let us repeat: this new method of attacking the problem is capable of giving dependable results. Our second conclusion, and the major thesis of the present investigation may be stated as follows: when graphitization does not occur during the process, the heating of pure iron-carbon alloys to temperatures above the eutectoid point produces in the metastable carbide phase a progressive diminution in carbon concentration. This diminution proceeds up to a temperature of approximately 900 degrees Cent. (1650 degrees Fahr.) and thereafter may reverse itself, although it has not been proven by the present investigation that this reversal is not due to graphitization involving a precipitation of atomic carbon too fine to be observed under the microscope.

Our third conclusion is that the A_{cm} line rises out of the $A_{3\text{-}2\text{-}1}$ line at a carbon concentration of 0.85 per cent, and that it is a linear function of temperature, at least up to 1060 degrees Cent. (1940 degrees Fahr.). Furthermore, when extrapolated to the eutectic temperature, it shows that fully saturated austenite contains 1.79 per cent of carbon. In this last conclusion our work is merely corroborative, though excellently so, of previous investigations.

This brings us to the point where speculations rather than conclusions are in order. The most obvious demand on our attention is made by the peculiar shape of the curve EKLFG. Schwartz suggests the following possible explanation: along the curve EKLF, cementite was breaking down according to the reaction,

$$Fe_3C = 3\ Fe + C$$

The atomic carbon was precipitated in an extremely fine state of division, probably colloidal or sub-colloidal. At this degree of dispersion, this carbon was held in solution, or if one chooses, in suspension in the residual carbide, rising to a maximum value at the point F. During this rise to maximum concentration, however, the precipitated carbon was coagulating into particles of ever increasing size until, finally, above the temperature of F, it began to precipitate out more rapidly than it was formed by the above reaction. This caused the carbide line to swing back into the region of lower carbon

concentration. The continuation GH of the curve FG has been somewhat arbitrarily drawn and is intended to suggest that, when visible graphitization does not occur, the carbide line may follow some such path. Schwartz's speculations are supported by some evidence. For example, at the point G in the high carbon specimen No. 5, isolated graphite does occur. Subsequent treatments at 1010 and 1060 degrees Cent. (1850 and 1940 degrees Fahr.) showed that these graphite particles were growing, both in number and in size, at a considerable rate. Even at the highest temperature, however, they were extraordinary with regard to their extremely fine state of division.

Just why certain specimens showed graphitization and others of almost identical nature did not, the authors are not in position to explain. It is probably a matter of that phenomena least understood, the formation of nuclei. Turning now to that second carbide curve which was derived, we find several points which demand explanation. First, why did the line change its direction at M and proceed toward higher carbon concentrations? The authors believe that the same phenomena which caused the curve EKLF to take the direction shown, are operative in the formation of the line MH, and that, in the absence of these phenomena, the line EM would proceed in the direction of the point S. The authors attempt no explanation of why these phenomena were delayed to such a higher temperature in the second case.

Attention should be called to the paper on "Graphitization of Prequenched White Cast Iron" by Schwartz, Johnson and Junge, published in the TRANSACTIONS of the American Society for Steel Treating, March, 1930. Here was presented the result of a single calculation similar to those of the present investigation. This calculation was made on a pair of specimens containing about 0.35 per cent silicon, and at a temperature of 980 degrees Cent. (1795 degrees Fahr.). The value of the carbon concentration of the carbide phase in this instance was 5.27 per cent. Taking into consideration the well known repulsion effect of silicon on carbon in iron-carbon alloys, this result checks very well with those of the present investigation.

In conclusion, the authors wish to call attention to the fact that few, if any, instances are known in which one metal exhibits absolute insolubility in another metal, and further, to the fact that solubility of a substance A, in a second substance, B, almost invari-

ably involves some solubility of B in A. Considering these two facts of physical chemistry, we can scarcely avoid the conclusion that it would be strange to find that cementite should be soluble to a marked extent in gamma iron but that, at the same time, gamma iron should be absolutely insoluble in cementite.

ACKNOWLEDGMENT

The experimental work here recorded was undertaken as an activity of the Department of Metallurgy of Case School of Applied Science in partial fulfillment of the requirements for the degrees of Master of Science in Metallurgical Engineering and Bachelor of Science in Metallurgical Engineering respectively by Bates and Lawson. The participation of Schwartz is limited to the suggestion of the problem and the method of attack, furnishing the Arsem furnace and special planimeter used and some consultation during the progress of the work.

DISCUSSION

Written Discussion: By R. H. Harrington, General Electric Research laboratories, Schenectady, N. Y.

The authors' use of mathematics, particularly the method of least squares, is very interesting and certainly to be encouraged. It would seem to have a possible application in prophesying the location of solidus lines, data for which is often scarce and difficult to obtain. Thus, a few data may be made to point the way with mathematical accuracy. However, for the location of such lines as the A_{cm} line on the iron-carbon equilibrium diagram it seems to the writer that a large number of actual observations by different methods are more nearly representative of the true situation. It is assumed, of course, that the calculation of the coefficients (in equations (1) and (2) on the fourth page) is correct.

The only apparent mechanical error in the method, as the writer sees it, is that some of the carbide in the quenched specimens may be of so small a grain size—perhaps submicroscopic—as to be incapable of measurement by means of the planimeter.

The writer agrees that the A_{cm} line is probably a straight line and offers thermal analysis data to that effect in the paper written jointly with Professor Wood[1]. The slopes of the two lines differ somewhat as might be expected since they represent the iron-carbon systems of two different series of iron-carbon alloys. The writer wishes to state emphatically that he accepts neither the A_{cm} line of the authors nor his own as deserving of *immediate* location on the Iron-Carbon Equilibrium Diagram. The work of a single investigation does not immediately become the standard for others. Especially is this true of

[1] R. H. Harrington and W. P. Wood, "The Critical Ranges in Pure Iron-Carbon Alloys", presented before the Twelfth Annual Convention of the American Society for Steel Treating, held in Chicago the week of September 22nd, 1930, printed on pages 632-658 of this volume of TRANSACTIONS.

equilibrium diagram work. The test of time and the judgment of other authorities are necessary. The authors' work, however, is of high correlative value.

Written Discussion: By W. P. Wood, associate professor of metallurgical engineering, University of Michigan, Ann Arbor, Mich.

I read this paper with much interest since it is concerned with a field in which I believe there is much interesting and valuable work yet to be done. We have been accepting the present iron-carbon equilibrium diagram as a more or less completed job but there is much evidence being accumulated which leads us to believe that we may have to modify our original conceptions concerning this system.

The application of the method of least squares to the relation between carbon content and temperature is appropriate I believe, but I wish the authors had given a more complete development of equations (1) and (2). I was not quite able to follow the relation of these equations to the ones presented by Schwartz, Johnson, and Junge in the previous paper to which the authors refer. The representation of the A_{cm} line by a first degree equation corresponds exactly to the ideas of Mr. Harrington and myself as presented in our paper appearing on pages 632-658 of this TRANSACTIONS.

The conclusions regarding the position of the "carbide" line as shown in Fig. 1, would have been more convincing if they had been supported by a larger number of complete determinations such as were carried out with heat numbers 5 and 19, locating definitely more points of the line ERMS.

S. L. HOYT[2]: This is an interesting attempt to clarify and improve our understanding of the iron-carbon equilibria. Any attempts in this direction are of course of extreme interest. The main object of my discussing this paper is to caution against too ready acceptance of the conclusions in this paper. The authors have indicated about the same sentiments. I believe they would be the first to recommend that we do not at once accept the Fe_3C phase as given by the paper. First of all the method is stated to be one which is susceptible to considerable accuracy, and the point which they raise in this connection, namely the verification of the A_{cm} line, is one which carries considerable conviction with it. The same determinations which presumably give this check on the A_{cm} line lead to carbon contents of Fe_3C, which are confusing. The first composition of this phase at 900 degrees Cent. is given at about 9 per cent (speaking from memory), and the checks on this same point lower this composition to somewhat below the usually accepted 6⅔ per cent to somewhere around 5 per cent. I attempted to figure out an explanation myself, but here the paper lacks the detail which really would be required either to substantiate this finding or to account for it satisfactorily. It seems to me that even in spite of the work which the authors have done it will be necessary to do considerable more work before we will be justified in changing our present conception of this particular phase of the iron-carbon alloys.

One question occurred to me as I was listening to Mr. Schwartz. In the paper a portion of it is credited directly to Mr. Schwartz in which he makes a statement to the effect that Fe_3C breaks down into iron plus carbon and follows that a little later with the statement which suggests that there is

[2]Research metallurgist, General Electric Co., Schenectady, N. Y.

some other mechanism for the formation of carbon. It seems to me that Mr. Schwartz has touched upon an exceedingly interesting point. I would like to ask him if he will not explain that just briefly for us so that it will be more clear.

O. W. Ellis[3]: I was interested in receiving a request to discuss this paper and wondered at the time I received it why I had been asked to discuss it, but as there were one or two points in connection with the paper which interested me, I thought that I might follow the request. I was especially interested in the curve in Fig. 1 representing the cusp at the point F, and the remark in regard to that curve which appears on the ninth page. This diminution proceeds to a temperature of 900 degrees Cent. and thereafter may preserve itself. There came to my mind when reading this paper something which may have nothing whatever to do with this, but which naturally comes to me through the association of approximate temperatures. Work was done by Saldan on the iron-carbon system, in which he investigated the variations in electrical resistivity with temperature, he found what had previously been found by another Russian investigator that in quite a number of cases there was a change in the electrical resistivity of the austenite at a temperature of approximately the 915 degrees that is referred to in Mr. Schwartz's paper. Again, in an investigation on the malleability of steel, which is reported in one of the Carnegie scholarship memoirs, the speaker noted that the malleability changed quite definitely in the austenite of steels at a temperature of about 930 degrees Cent. Some little while prior to our investigations on the malleability of steel, Andrew Rippon and Wragg, as a result of experiments on the effect of rate of cooling and initial temperature on the critical points in steels, arrived at the conclusions, which may or may not be right because, of course, at the present time investigations in regard to these alloys are being subject to change, that the iron carbide in austenite was subject to dissociation with increase in temperature above the critical range and that as a result changes might occur in austenite, such as they actually found in their dilatation experiments at about 930 degrees Cent. This, of course, is an idea which was put forward long ago by Professor E. D. Campbell. The point which I wish to emphasize here is that there does seem to be evidence that changes occur in the iron-carbon alloys at temperatures in the neighborhood of 930 degrees Cent. and that these changes are worth investigation and worth taking note of. Whether they have anything to do with the changes that have been described by Mr. Schwartz is another matter. Whether the points that have been observed are due to experimental errors on the part of those who have carried out the different types of work, I do not know, but it is certainly interesting that no less than three investigators have noted some discontinuity in the behavior of these alloys at about the temperature that is shown in Fig. 1 of Mr. Schwartz's paper.

Authors' Closure

I think that the viewpoint which was brought out, particularly by Dr. Hoyt, is eminently proper. I have no idea that Mr. Bates and Mr. Lawson

[3]Director of metallurgical research, Ontario Research Foundation, Toronto, Ont., Canada.

had any suggestion that we were at once to introduce these lines in the iron-carbon diagram. I think, however, that it is equally important, and I think there is enough evidence to say that we have to stop drawing a vertical line at Fe_3C. The desire for more observations is quite understandable. The explanation as to why they were not available is simple, as those gentlemen who are teaching will know. This was a thesis subject and the time came when the investigation had to be closed and the amount of work which was recorded is the amount of work then accomplished. The difference between values is not as great as Dr. Hoyt apparently assumed. There are two different pairs of alloys represented in that investigation. One pair of alloys gave the cusp running toward very high carbon, and the other pair always gave that curve which has a bend toward the left. We know that the one which gave the cusp will graphitize on prolonged heating. They were both pure iron alloys, that is as nearly pure as one could make. The reason why one graphitizes and the other does not is tied up with a thing we have been struggling with for 10 or 12 years, the variation in graphitizing rates. We observe that the one which tends to graphitize makes the cusp before it graphitizes and the other makes the other line. A considerable time interval elapses during the graphitization of white iron during which the amount of carbide decreases but no free carbon appears.

There is one other item on the question of how reliable the answers are. Observation yields two results, one for A_{cm} and one for the carbide line. They are mathematically connected, and I do not see how one can be wrong and the other right. The possibility that one may miss sub-microscopic cementite in quite probable. If they had missed such carbide, the austenite composition would have come out higher than the A_{cm} concentration. With such limits of error as indicated it did not come out higher so that is the only experimental proof I know as to the elimination of errors due to not being able to see minute carbide spots.

Our usual belief is that we have not ever actually observed the direct breaking down of carbide in a single phase alloy. What usually happens is that reaction takes place in a series of steps which involve the solution of iron carbide in some form in gamma iron and the crystallization of carbon from that solution as free carbon. We have made diligent attempts to see cases where that should not be so, but we have never yet succeeded in doing that. The suggestion might be made in connection with this paper that what happens is that when a few atoms of carbon are formed by the decomposition of cementite, perhaps those atoms dissolve in other cementite to make a saturated solution of carbon in cementite whereas ordinarily they dissolve in austenite. The austenite, being supersaturated, crystallizes carbon out of itself. Those two mechanisms might be possible.

A STUDY OF THE GRAIN STRUCTURE OF MARTENSITE

By K. Heindlhofer and E. C. Bain

Abstract

This paper offers a few data relating to the crystalline structure of martensite. Two principal methods of study were employed, X-ray diffraction and examination with the petrographic microscope. A portion, which may seem disproportionately long, of the paper is devoted to an accurate description of X-ray diffraction and the mode of its interpretation. This has seemed necessary since the value of the data depends almost entirely upon the control of the conditions of the experimentation and upon the interpretation of the results in terms of atomic structure.

From their studies the authors draw the conclusion that martensite is chiefly composed of characteristically distorted ferrite "units" or grains the size of which is not greatly different from that of the parent austenite. The type of distortion is principally that developed by twisting or bending, in such manner as to retain a moderate constancy of spacing between the 1 1 0 planes but at the same time to vary the orientation of some of the same planes through a considerable angle. In addition the ferrite units possess a marked random atomic irregularity of the "solid-solution type."

The hypothesis is submitted that the hardness of martensite is not explained on the basis alone of the grain size or of any distortion of the cold-work type (which is definitely present) but more probably depends upon the strong interatomic bonding within the greatly supersaturated pseudo-solid solution of carbon in ferrite.

I. Introduction

THIS paper does not offer any new theory of the hardening of steel or the nature of martensite but confines itself to the presentation of a few observations which are thought to be pertinent and of the hypotheses to which these observations lead. Before

A paper presented before the Semi-Annual meeting of the society held in New York City, February 7 and 8, 1930. The authors are members of the society and also members of the staff of the research laboratory of the United States Steel Corp. E. C. Bain is metallurgist and Dr. K. Heindlhofer is physicist. Manuscript received January 16, 1930.

setting forth the data and conclusions of the present investigation a statement of the viewpoint from which the problem was approached may be justifiable. The viewpoint was maintained also in interpreting the results and in the account of the enquiry.

The study of the nature of martensite formation in steel has engaged the best effort of metallurgical research laboratories of late years and clearly much information of great importance has been offered. The viewpoint of the present authors in this investigation relates to the interpretation of some of these items of information, or more particularly to the weighing of some of the evidence. It has seemed that the brilliant experimental work of research metallurgists should serve to permit chiefly the gradual rejection of *some* of the theories offered for the hardness of quenched steel *rather than* the exclusive establishment of any one of them. The knowledge in their opinion is not complete enough to warrant the construction of a theory, particularly any simple one. The evidence forcing this opinion is almost unmistakable from a review of the whole of the literature, but the recent assembly of opinion by Sauveur[1] serves to indicate both the fair agreement on some few points and the divergence of opinion on most other points. Presumably some of the evidence used in support of the various theories has been either fundamentally incorrect or incorrectly interpreted, but in the main there are probably not enough data.

In connection with this investigation, before any experimental work was inaugurated, the "data of martensite" was reviewed and critically examined on the basis of three criteria: 1. Is the item of information offered as evidence a direct observation or one arrived at by "reasoning by analogy"? 2. Is the item necessary to the establishment of some theory or, instead, an interesting related observation only possibly germane to the subject? and 3. Can the item be checked with any further accuracy in the time available for this investigation?

As for the first consideration, data adduced by analogy were entirely rejected from the evidence. The science of metals may no more be justified in the misuse of analogy than the science of physics, for example. Analogy is of extreme usefulness in determining the trends that a research should profitably take and

[1]Albert Sauveur, "The Current Theories of the Hardening of Steel—Thirty Years Later." *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 73, 1926, p. 859.

for the visioning of temporary hypotheses but is of course no evidence at all in the *establishment* of a theory. The second consideration is one more difficult to use and is here very conservatively applied lest significant information be unrecognized. The third basis led the present attack to a study of the grain structure of martensite, since it is not by nature a matter of "reasoning by analogy" at all, is certainly most vital to the establishment of some of the newer theories, and as an investigation could effectively be carried out in the authors' laboratory.

The list of possible problems which would lead to results needed in the construction of a complete picture of the hardening of steel was almost identical with some of the list of questions proposed by Sauveur[2] in his recent logical critique of martensite theories.

II. The Methods of Study

The metallurgical microscope in the hands of such men as Lucas and Hanemann may well be regarded as having yielded all that may be hoped for without the unexpected circumstance of some revolutionary development. The technique of the microscope has been perfected far beyond the present status of X-ray crystallometry. It may be assumed that in the flawless photomicrographs at the limit of magnification compatible with the fundamental resolution of our best optical systems, as much detail of etched martensite has been seen as is at present possible or perhaps as will be possible for some time. The microscope has been helpful in our conception of martensite but not decisive as to any particular theory of the mechanism of martensite formation.

The X-ray equipment available had been designed to make crystal studies of greater accuracy and refinement than had hitherto been made, so far as is known. It will be shown that much of the work with X-rays has been slightly less significant than was thought and in some few cases distinctly less trustworthy when attempt has been made to evaluate quantitatively imperfections in crystallinity. This applies to some of the earlier work of the present authors who have attempted in this investigation to subject their methods to such criticism as would result in bringing X-ray technique to a condition more nearly comparable with other precise measurements. In particular, the refinements in X-ray crys-

[2]Albert Sauveur, "The Decomposition of Austenite." Transactions, American Society for Steel Treating, Vol. 17, 1930, p. 199.

tallometry involve more precise knowledge and control of true specimen size and parallelism of the effective beam.

The first method of study then was by means of X-ray diffraction. The second principal method was by means of the petrographic microscope utilizing polarized light and crossed Nicols. In this attack the authors were particularly fortunate in having the advantage of the valuable advice of Dr. Olaf Anderson, petrologist at the U. S. Steel Research Laboratory. As will be seen, the results of the two methods of study are mutually corroborative.

III. The Study by X-Ray Methods

A. Preliminary

There seems to be complete agreement among all reports that the Hull method has served to demonstrate that martensite is alpha-iron with some characteristic imperfection, distortion, or crystalline idiosyncrasy which is reflected in its X-ray diffraction pattern. Furthermore, with high carbon and as quenched from the higher temperatures, hardened steel may be composed of some crystalline entity which is not precisely cubic but may reach a departure from cubic symmetry represented by a tetragonal system with an axial ratio as great as 1.06. Most theories advanced have ignored this interesting circumstance completely, presumably because heating to a comparatively low temperature causes it to disappear (changing into the more nearly cubic ferrite) without substantially softening the martensite. However, a complete picture of the mechanism of hardening must recognize the tetragonal martensite. The Hull method has been very effective in demonstrating these basic facts and is almost perfectly suited to the accurate determination of lattice constants in well crystallized substances by the use of a comparison substance with known lattice constants. It is, however, very badly adapted to the study of imperfections of crystallinity in a specimen. It has, therefore, been used only for one or two purposes and instead the more particularly adapted pin-hole method has been employed.

The Hull Method

The Hull[3] method is characterized, 1 by employing a wide and flat effec-

[3]A similar method described independently at about the same time is known as the Debye-Scherrer method. Any difference between the methods would lie principally in the shape of the specimens. The Hull specimen is the long slender cylinder while the Debye-Scherrer specimen is usually a smaller one of circular contour.

tive beam of characteristic X-rays radiating a relatively long, cylindrical polycrystalline specimen (not usually a circular cylinder) whose axis is perpendicular to the direction of the beam and in the lateral plane of the width of the X-ray beam, and 2 by employing a curved film encircling the specimen as a part of a circular cylinder whose axis is the axis of the specimen radiated.

By reducing the true specimen extent for any given diffraction angle to a mere thread a few thousandths of an inch in width (normal to the diffracted beam) diffraction lines of great precision may be obtained enabling lattice measurements with an accuracy in the vicinity of one part in one thousand to be made, with every precaution being taken and a simultaneous recording from an equivalent specimen of a material of known parameter. Often too great accuracy has been assigned to lattice constant determinations.

It should perhaps be pointed out that in reality a fine-grained specimen of well crystallized material in the X-ray beam produces a Hull crystallogram which is the photographic recording of two possible infinitudes of diffraction images. The Hull crystallogram line is in reality the photographic record of a purely statistical maximum of a very great number of diffraction images which, in all, may cover a very wide band on the film. Any tiny crystal in the radiated specimen is the potential "reflector" of a target image in any direction limited by the elements of one cone whose axis is the line connecting the crystal with the target, and whose apex angle is the fundamental diffraction angle θ ($\sin\theta = \frac{\lambda}{2D}$). As the crystallite is revolved on this axis the locus of its diffraction images is the intersection of the cone with the film. With fine-grained substances (which make the best samples for the Hull method) nearby crystal fragments are certain actually to direct radiation in many points on such a cone. But with the long specimen of the Hull apparatus very remote crystallites (usually the specimen may be about 10 millimeters long) will diffract X-rays along elements of such cones quite divergent one from another. There are then an infinite number of such cones between these limits imposed by the length of the specimen which at their intersection with the cylindrical film constitute the loci of possible overlapping diffraction images. Obviously then each line in a Hull pattern is the *envelop* of an infinite number of curves.

When a substance is not composed of a sufficiently large number of crystal fragments to render the images entirely over-lapping this family of cones is actually apparent as in Fig. 1A. To clarify this complicated analysis of the geometry of the formation of the Hull crystallogram a chart, Fig. 2, is offered which shows how these families of cones intersect the film and record the envelop. It will be seen that in no case is this envelop a straight line, although for the small angle lines from the greater spacings it closely approximates a straight line. This circumstance may account for the error in some lattice constant measurements described, for it is usually not recognized that this curvature of the envelops (and hence the crystallogram lines) is inherent in the method. The chart, Fig. 2, is drawn with the scale in the direction of the increasing angles elongated 10 times.

But what is more important, the single separated images of the target diffracted to the film may lie remote from the envelop which should be



Fig. 1—Hull Pattern of A Ferrite, and B Martensite.

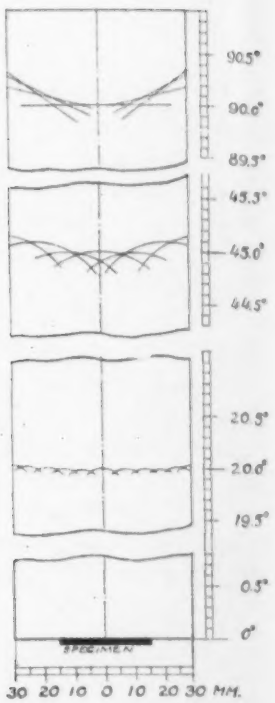


Fig. 2—Schematic Hull Diagram Assuming Point Source, 30 mm Long Line Specimen, Distance from Source to Specimen 200 mm, Radius of Film 200 mm, Longitudinal Scale × 10

measured. Since, of necessity, it is only when the statistical radiation maximum indicating the location of the envelop is itself a very narrow distribution curve of intensity that angle measurements are accurately made, the method inherently fails when the number of total diffracting grains and images is not very great. It is then ill suited for any study of a few grain fragments even when the specimen is ideally chosen or even if only lattice constants are being studied.

However, when the precise limitations of the method are well understood the method offers an opportunity for qualitative determination of grain size over certain large ranges.

A. When very few (hence, usually, very large) grains are present the film records only a few separated diffraction images of the target. These images may be sharp or diffuse, depending upon the equipment and the crystallinity.

B. With medium grain size the statistical effect begins to be apparent and fairly smooth bands are formed whose position is well marked provided the active specimen is small. This is the normal case with properly annealed metals of suitably fine grains.

C. When particles of colloid size exist the individual images are each somewhat diffuse and the bands are continuous and smooth, but slightly widened. However, this is of little significance in most cases since the broadening caused by any such fine particles is still less than that caused by the usual combination of target size (as viewed from the specimen) and effective specimen size. This point will be considered in more detail under the discussion of the single diffraction images. The widening of lines in the Hull method caused even by slight deformation of normal sized grains far exceeds that caused by the colloidal materials reported or examined by the authors.

There is one circumstance which makes this qualitative manifestation of diffuse diffraction bands in the Hull pattern easily recognizable. It is customary to use only the alpha radiation from the K series of characteristic spectral lines of the target metal for the Hull method. This alpha radiation is never a single wave-length, but two distinct wave-lengths, each of which is capable of creating its own crystallogram pattern. The two component wave-lengths are very close together (for molybdenum, for example, .708 and .712 Angstrom Units (cm. x 10^{-8}). Thus if conditions of the diffraction are such as cause (1) the images of the target to be diffuse or (2) the envelop to be wide, the two distinct patterns tend to merge, and there is said to be no "resolution of the alpha doublet". The separation of the two patterns is wider in the high diffraction angles and hence the "resolution" is more easily perceived in those bands. Some investigators have evaluated the confusion in terms of the lowest angle at which the separation was visible. However, this entire effect is quite as much dependent upon the size and shape of the specimen and the target size as viewed from the specimen as it is upon the nature of the specimen, hence such results are only valuable when the method is one of comparison between equivalent specimens or wherein all constants of the equipment are carefully evaluated and recorded. With properly made specimens of well crystallized material all the crystallogram lines are "resolved". The specimen to be studied for diffuse

diffraction should be identical in contour with a specimen which had shown such resolution.

The degree of confusion in the sharpest part of a Hull crystallogram line is dependent upon:

1. Size of target (as viewed from specimen),
2. Size and contour of true specimen,
3. Distance of specimen from target,
4. Distance of specimen from film; *as well as*
5. The degree and nature of the imperfections of crystallinity, or
6. The minuteness of the grain size.

Furthermore, in practice, the evaluation of the confusion of any diffraction pattern or image is dependent upon a correct length of exposure, to prevent insufficient total exposure from narrowing the apparent extent of the darkening and to prevent over-exposure from unduly widening by halation the apparent darkening. The significance of any observation is only in proportion to the completeness of control and recording of all of these factors. All the foregoing statements regarding the Hull method are demonstrable by purely mathematical considerations of electromagnetic theory of radiation and the geometry of the equipment.

Since the present study deals with the grain structure of martensite only the last two items are of interest, the grain size and degree of crystalline imperfection. Widening from other causes must be controlled or eliminated. It has been computed that the increase in widening of the crystallogram bands with increase in diffraction angle (with decrease in inter-planar distance) follows very different functions in the two cases, colloidal sized grains and distorted grains. The increase in widening for colloidal size is not very rapid with the great angles—only about in proportion to the secant of the half-angle. For the widening caused by elastic unsystematic distortion of the normal grains the widening is so great that even for an infinitesimal sample the bands may include four or five degrees at $2\Theta = 90°$. Hence in a qualitative way very fine grain size may be distinguished from large but distorted grains. The proof of the distinction may be visualized when it is considered that a given displacement of any few atoms from the regular geometric arrangement in the lattice may constitute a negligible warping of the wide spacings, but for the narrow spacings may constitute in the same position a very large irregularity approaching the half-way position for *total extinction*. Thus the falling off in apparent intensity (accompanying the broadening) of successively larger angle lines in the Hull pattern is extremely rapid for distorted grains (badly crystallized material) and only slight for fine grain size (colloids).

The fundamental advantages of the Hull method when applied to those investigations for which it is suited are important. They consist (1) of the short exposure permitted by the use of a large number of contributing crystals and a close approach to the target which is fore-shortened into a line source, thin in the proper direction, and (2) the nice accuracy of line definition when a proper sample is employed, and (3) the ease of linear angle measurements, and (4) the absence of exaggerated falling off in intensity for high angle diffrac-

tion on a flat photographic film in which the intensity follows the $\frac{1}{\sec^2}$ relation.

On the basis of the limitations detailed above of the Hull method, Hull patterns were made of the martensite used in the investigation—both the thin material and the more massive type—for the purpose of comparing the results with the pattern derived from colloidal material. The crystallograms were in no wise unlike those presented by many investigators, including the authors. See Fig. I B. As usual the intensity fell off with higher angle (smaller spacing) diffractions so rapidly that at $2\Theta = 90°$ the bands were too weak and diffuse to be apparent. This decrease in intensity is many times more rapid than permissible with colloidal particles. Hence a reasonable doubt was established of the theory that martensite is fine-grained, and the possibility of merely distorted (permanently or elastically) grains was established.[4] These results confirmed the necessity for the use of the pin-hole method for more decisive results.

The Pin-Hole Method

There is very little difference in mode of fundamental X-ray diffraction between the Hull method and the pin-hole method, aside from the geometrical differences in appurtenances and in the distribution of the elements of the equipment. If the cylindrical film of the Hull method were replaced by a flat plate normal to the X-ray beam the full cones of the diffractions would fall upon it and record as approximate circles. It would be found that these circles were sharply and narrowly defined on the plate at its intersection with a plane through the center of the target, and perpendicular to the specimen axis, but very diffuse at right angles to this intersection line. The very circumstance that constitutes the advantage for the Hull method causes this condition and it is eliminated in the pin-hole method by the use of a small specimen preferably circular and the elimination of the flat beam of X-rays. While it is customary to use a thin specimen through which all scattered rays are "transmitted" the method of "surface reflection" by an incident beam may also be used, except where the circles of the crystallogram are reduced to semi-circles (approximately). In case grain-size determinations are to be made the latter device is not well adapted since it is almost impossible to estimate what volume of the specimen participated in producing images. With a thin transmission specimen the active volume may be measured directly if the specimen is smaller than the pin-hole, or the effective specimen volume may be computed in case it is, in all, larger than the pine-hole.

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[4] H. Mark, "The Determination of Particle Size by the Use of X-Rays." *Transactions*, Faraday Society, Vol. XXV, July 1929, p. 387.
"Ueber die Verbreiterung der Debyelinien bei kaltbearbeiteten Metallen," by U. Dehlinger, *Zeit. für Kristallographie*, Vol. 65, 1927, p. 615.

A very effective method of grain counting can be carried out by means of the pin-hole diffraction camera. The actual accomplishment is quite simple, although the science of the method is somewhat complex. It is necessary first to review the fundamentals of diffraction by white radiation.

In every crystal diffraction is possible from any system of parallel planes. In the crystal there may be a number of such families of planes which are identical in spacing. Thus, for the crystal of alpha iron (martensite) there are in all six families of 1 1 0 planes identically spaced. If such a single crystal were rotated into all conceivable positions all of these six plane families would record diffraction images ultimately on the pin-hole film; all images would lie in a single circle for any single wave length of X-rays. Only one image would ever result from a single position, but each of the six families would produce an image when in its particular position. With two different wave-lengths complete rotation would produce two circles, etc. Further, if the wave-length were varied continuously from an extremely small value up to twice the specific interplanar distance of the crystal, there would be one diffraction image recorded for each of the six families. That is to say, if the fixed crystal contained only these six families each capable of forming a diffraction image, and were surrounded by a spherical film, then after exposing the sample equally to all these wave-lengths of X-rays, the spherical film would record *just six* first order diffraction images of the target. If three such crystal grains, at random orientation, were present then we should find just 18 images—and so forth. If half the spherical film were cut away then we should find half the number provided the total number of grains were sufficient to render the results statistical. This is the basis of a method presented here for grain counting by the pin-hole diffraction method. However, the modifications are very important since the basic principle includes a vast number of practical difficulties which may be overcome.

The final method consists of counting the number of target diffraction images which are touched by a single line circumference drawn on the flat film with the direct beam spot as center, and multiplying this figure by a constant for the particular set up. It is important that the region wherein this circumference is drawn contain only first order diffraction images, and that it contain likewise no images from spacings other than the one used in the consideration. This is accomplished by controlling the range of white X-rays used so that no such extraneous images could be recorded and yet the range must be great enough to encompass the full images from all grains properly located. The constant referred to is obviously just a function of the radial width of a single image, or more properly the angle included by the extreme edges (radial) of an image. The constant actually is a number, 180 degrees divided by the image inclusion angle in degrees. The image inclusion angle is most properly measured on a good crystallogram, but may be computed for use with perfectly crystallized substances. In the latter case the inclusion angle for all practical purposes is the sum of the angle subtended by the target as viewed from the specimen plus the approximate angle included by each grain as viewed from the target. (The latter is, of course, very small). The method is reliable for all magnitudes of grain count which on the one hand are sufficient to behave

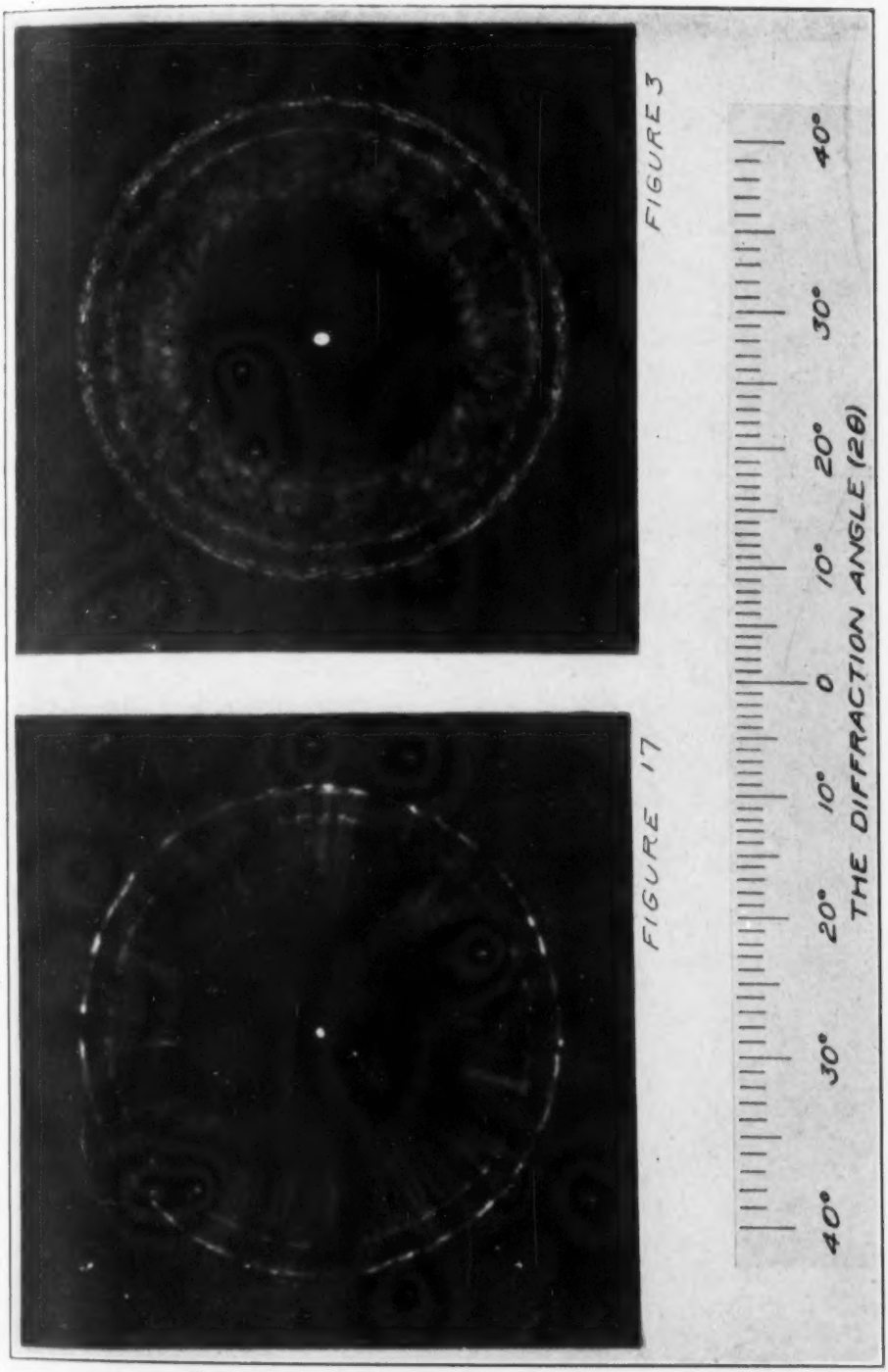


Fig. 3—Pin-hole Crystallogram of Well-crystallized Eutectoid Steel.
Fig. 17—Crystallogram of Tape, Carbide Spheroidized, Elongated to Rupture, Sample Chosen Very Near Fracture.

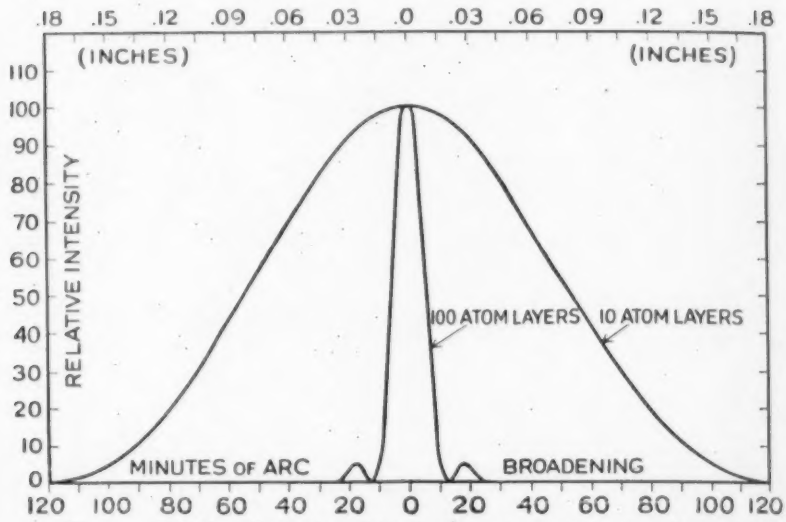


Fig. 4—Relative Intensity Distribution of the Mo Kα Rays Diffracted by a Family of 1 1 0 Planes of a Single Colloidal Ferrite Grain Around the First Order Diffraction Maximum as Calculated.

statistically and not so numerous on the other hand as to cause excessive overlapping and complete loss of separate images as in the case of colloidal particles.

Fig. 3 is an example of such a crystallogram of medium grain-size material wherein each crystallite is very well crystallized. It will be seen that in the region inside the characteristic circles there are numerous well defined images of the target whose size depends upon the angle mentioned above. The image of the target is widened (radially) slightly due to the size of each diffracting grain, but this is a very small magnitude. This specimen was of eutectoid steel wherein the carbides were very well spheroidized into rather coarse units. Grain-size estimation of the ferrite by means of the microscope is practically impossible in such materials. Scattered among the images caused by ferrite grains are, no doubt, images from the cementite crystals.

Grain fragments in metals are usually very imperfectly crystallized, and are incapable of producing such well defined images of the target. As has already been mentioned, very tiny colloid fragments, even though perfectly crystallized, also fail to produce sharp images. This is merely the manifestation of a well known principle of diffraction. In Fig. 4 the actual distribution of intensity within the principal region of the diffraction "spot" is plotted for a fragment of ferrite in which 10 and 100 atomic planes participate.[5] The planes chosen are the 1 1 0 planes and the chart applies to the first order diffraction of the molybdenum K alpha X-rays. The source of X-rays is assumed to be a point. The actual entire width of the diffraction band (or circle) on a crystallogram would be wider by the value of the angle included by the target as viewed from the specimen plus the angle of the entire specimen as viewed from a point on the target. This angle increment would, of course, be interpreted as an radial intercept on the crystallogram before being deducted.

[5]The computation of the distribution of energy over a diffraction spot from a few diffracting planes was originally appended. However it seems sufficient to refer to an article since published; Murdock, C. C., "Form of the X-ray Diffraction Bands for Regular Crystals of Colloid Size." *Physical Revue*, Jan., 1930, Vol. 35, p. 8-23.

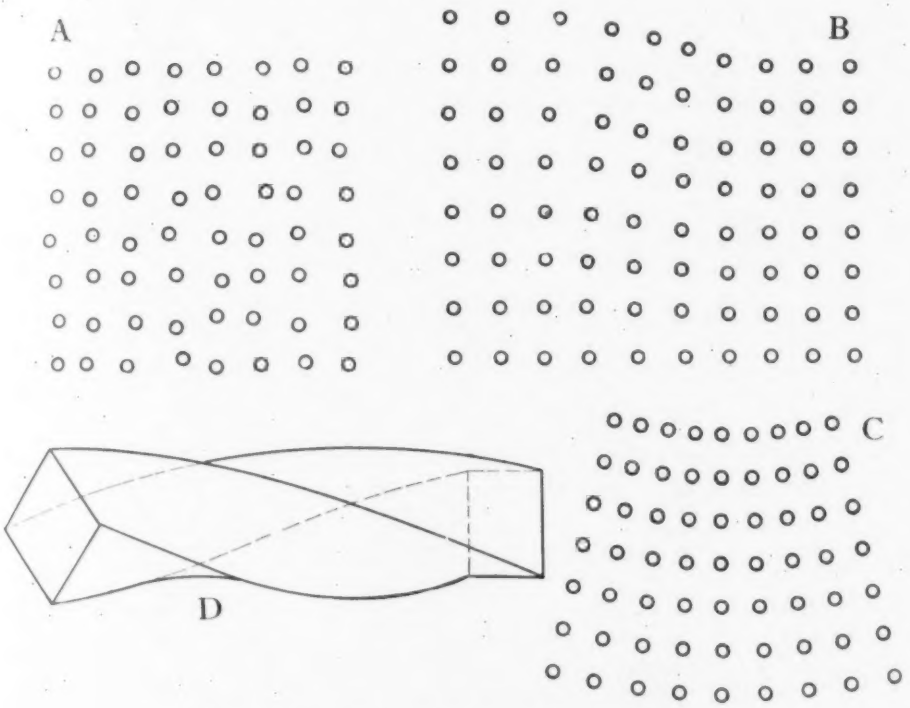


Fig. 5—Various Types of Lattice Imperfections.

The diffusion of a single image caused by a single grain may be due to several types of crystalline imperfection. A few are sketched conventionally in Figs. 5 A, 5 B, 5 C, and 5 D.

Fig. 5 A. Random staggering of atoms in the planes such as might be caused by the presence of foreign atoms in solid solution has only the effect of weakening the intensity of single diffractions and hence of the bands or circles in polycrystalline patterns. The weakening is, of course, much more marked in high angle diffraction where the distortion amounts to a more serious impairment of the grating. The widening is negligible in comparison to the weakening.

Fig. 5 B. Systematic distortion of a crystal in such a manner as indicated results only in a slight widening of a single image caused by white radiation, but the image locally is of low intensity. Such distortion greatly weakens the intensity of its monochromatic diffraction with some widening.

Figs. 5 C and D. Bending and torsional stresses below the magnitude causing permanent slip are represented schematically as producing the distortion. The effect would be quite similar probably if slight permanent slip had occurred at various points. Such distortions manifest themselves by extreme radial widening of single diffractions of the white radiation but by very little, if any, widening of single images formed by monochromatic radiation. This type of image confusion was found for the martensite. In every case the confusion discussed refers, of course, to that in excess of what may be caused by the apparatus with perfect crystals.

B. Material, Apparatus and Procedure

In proceeding with the study of the grain structure of martensite it was clear that a means of studying the imperfections of crystal fragments would be required in addition to checking the preliminary evidence against mere fineness of crystal grain. If representative martensite were composed of sufficiently large crystal fragments then it should be possible to isolate one and study it by means of the original Bragg method in which images of the target could be recorded quickly on a film (or even observed on a fluorescent screen), and from the nature of this image its crystalline imperfections, if any, deduced. The nearest approach to this desideratum was the use of as small a sample as would suffice and its examination both by "white" radiation and some kind of monochromatic radiation (characteristic spectrum line). Each radiation would serve a purpose in reducing the number of possible types of imperfection to a final limited choice. With "white" radiation a single crystal (the Laue method) would almost certainly produce images in any position it might occupy in the beam. The distribution of energy within the image would depend upon the factors already discussed in connection with the Hull method with a few modifications, all of which are entirely capable of complete analysis on well-founded radiation theory. With monochromatic radiation a fair number of crystals must be present to insure the high probability of any image at all being diffracted, or else the single crystal should be rotated.

As it happens, the pin-hole method of X-ray diffraction realizes all these conditions to a satisfactory compromise if the specimen contains a certain order of magnitude of grains. But the inferences which may be drawn depend wholly upon the usual constants of the equipment, the target size, the specimen size, the distances separating specimen from tube and from recording film. The pin-hole method requires a small including angle of target (usually accomplished by an unusually long distance between tube and specimen) and a flat film of sufficient size to include the entire cone of possible diffraction from the very small specimen which should have its plane precisely normal to the main X-ray beam. The character of the X-rays may be either monochromatic or "white" radiation (as desired in the Laue method using a single crystal) or both may be employed. The last type was most suitable for this investigation. The effective target, as viewed from the specimen, should preferably be not an

elongated ellipse, but a circle; in the present equipment the target appeared as wide ellipse, the minor diameter being .7071 (sin 45°) times the major diameter. The distribution of energy as radiated from the effective target is shown by the pin-hole photograph, Fig. 6.

The Material. Eutectoid carbon steel was selected as being the most representative metal and also because of its having the least tendency to introduce any second constituent into either the austenite

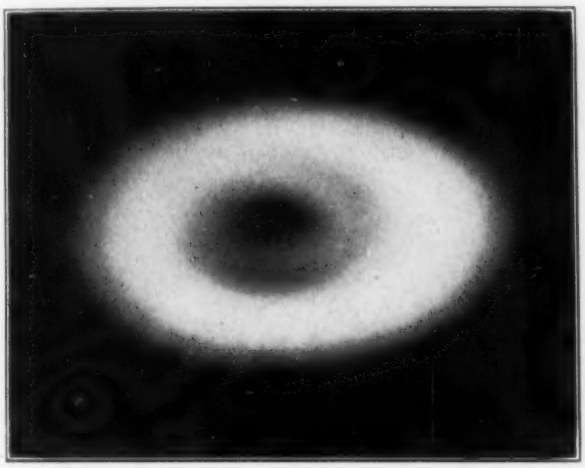


Fig. 6—Magnified Pin-hole Picture of Target.

as formed or into the martensite during quenching. The ideal thickness for a pin-hole sample examined with molybdenum radiation is about 0.002 inch (0.05 mm.). The preparation of samples of this thickness from large pieces of quenched steel is a difficult procedure, and it was therefore decided to utilize, if possible, strip steel of approximately this thickness. To maintain the carbon content the strip was heated (in a fixture) in cast iron chips. (Original carbon 0.96 per cent; after treatment 0.98 per cent). To develop a suitable austenite grain size it was found that about 45 hours at 950 degrees Cent. (1760 degrees Fahr.) was required. It was found possible to trace the austenite grain boundaries without their being marked out with excess cementite. A process of deep etching followed by repolishing and lightly re-etching produced the appearance seen in Fig. 7, from which austenite grain counts could be made. To be certain that the results were not unlike those obtainable from massive pieces of steel one specimen about 0.0625 millimeters (0.0025 inch) in thickness was prepared by grinding under water. Its crystallogram is shown in Fig. 8 for comparison with the crystallogram of the strip

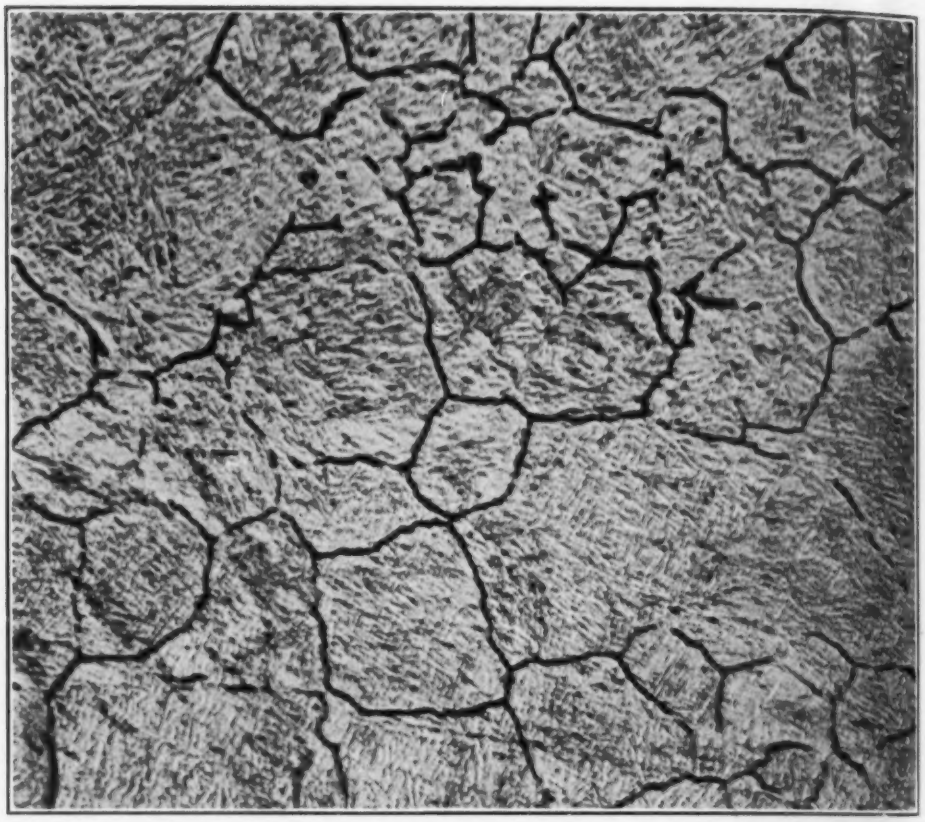


Fig. 7—Photomicrograph of Martensitic Specimen Specially Etched to Show Austenite Grain Boundaries. × 500.

steel. Fig. 9 shows the martensitic structure characteristic of all the samples examined. The austenite grains were not excessively large, and were surprisingly well equiaxed as shown by examining the microstructure of the strip on end and side. Some satisfactory tools have possessed nearly as coarse austenite grains. It was believed that the value of the experiment would be greater if conditions as nearly normal as possible were maintained. To accomplish as much destruction of austenite as possible the specimens were refrigerated repeatedly to liquid air temperature. This treatment, however, produced no visible change in the spectrograms. The specimens were also tempered at various temperatures. The constants of the apparatus are as follows:

The light-proof film container, the accurate spacer, the lead pin-hole and the collimator, Fig. 10 (if used), constitute the pin-hole apparatus, and, except as otherwise noted, each has the following principal constants:

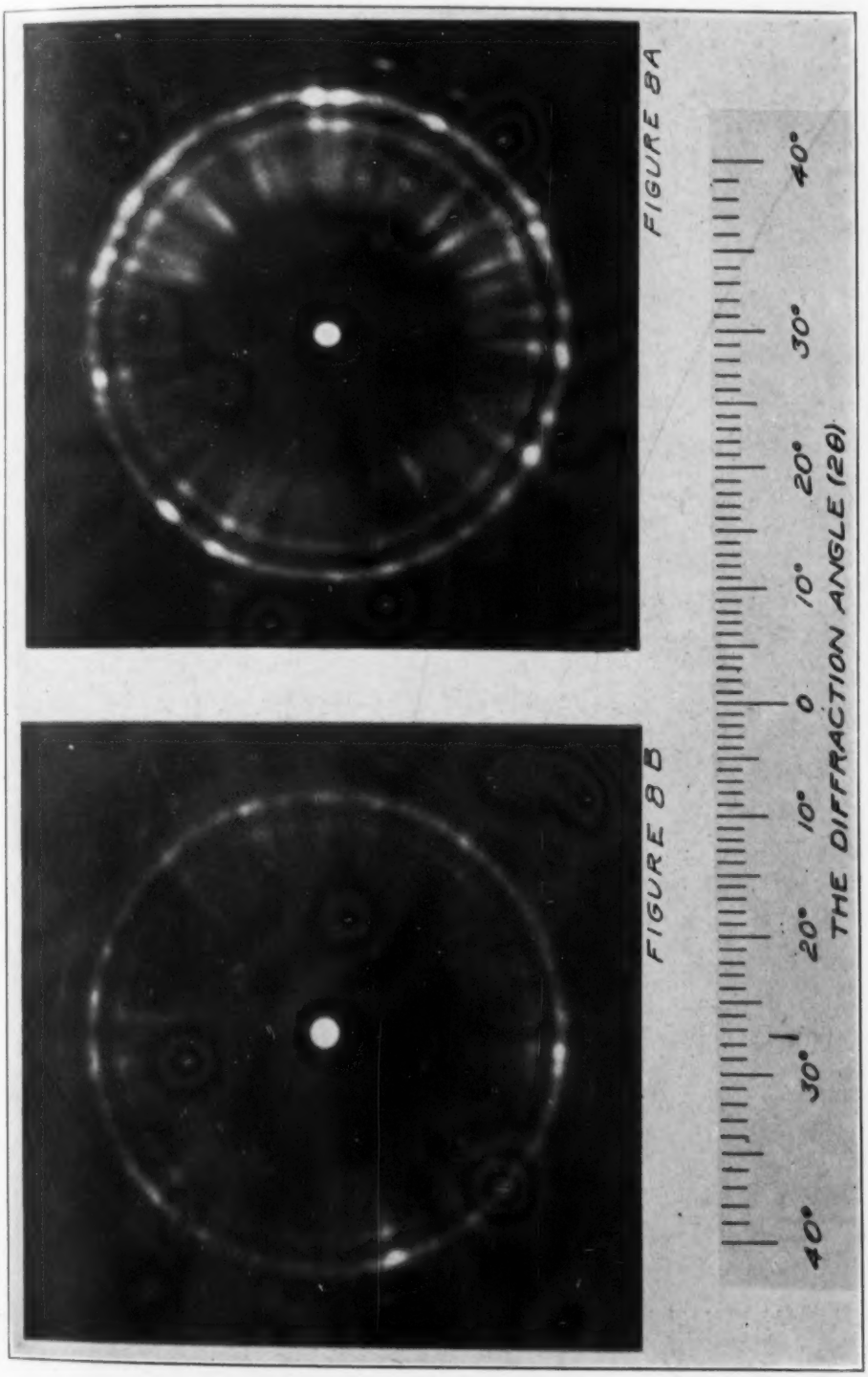


Fig. 8—Pin-hole Crystallogram of A, Massive Martensite Ground to 0.06 mm. Under Water, in Comparison with B, Strip Specimen, Both from Large Specimens.



Fig. 9—Photomicrograph of Strip Martensite at × 2100.

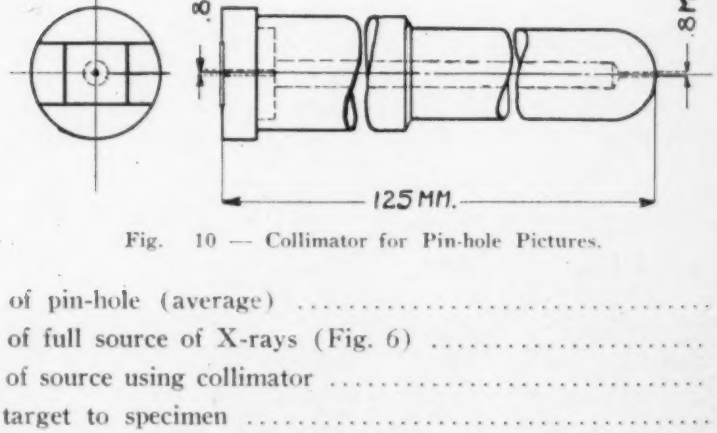


Fig. 10 — Collimator for Pin-hole Pictures.

Diameter of pin-hole (average)	0.4 mm
Diameter of full source of X-rays (Fig. 6)	4.8 mm
Diameter of source using collimator	1.6 mm
Distance target to specimen	270.0 mm
Distance film to specimen	50.0 mm
Angle of inclusion of source (at specimen)	1.0°
Angle of inclusion of source, with collimator (at specimen)	0.4°
Angle of entire effective specimen (at target)	0.2°

From the constants listed above the confusion of diffractions (radial) may be computed in so far as they result from the apparatus alone, i.e., with perfect crystals. Such radial distances on the attached scale are as follows:

Radial distance of each image (white radiation) full target	1.0°
Radial distance of each image (white radiation) collimator	0.4°
Width of monochromatic circle of images (total)	0.6°

From the total width observed, these values must be subtracted to determine what degree of broadening is contributed by imperfections of crystallinity. It should be clearly understood that the width of the monochromatic ring or circle is, with perfect crystals, the intercept of the target angle plus the projection of the specimen diameter, but there is no error in giving these quantities a numerical value in degrees or arc. when the scale is used. The use of such nearly parallel X-rays is regarded as essential to an accurate study.

It was thought advisable to obtain the pin-hole crystallograms of the strip martensite as tempered at various temperatures using, in every case, the identical portion of metal—with the same original austenite grains. This involved attaching the specimen to the brass collimator rather than to the lead pin-hole. The only method which would still bring the specimen in firm contact with the pin-hole (a necessary condition) seemed to be the use of a very shallow bezel. This was really practicable, and it is believed that the specimen remained entirely immovable with reference to the collimator which is illustrated in Fig. 10. The strip was retained in close contact with the lead pin-hole by a set screw which engaged the collimator in an accurate hole in the camera casting.

The metal used for the martensite specimens was cold-rolled after some reduction by wire-drawing. It exhibited therefore the condition of preferred orientation of the ferrite grains. The preferred orientation was of a type intermediate between that of wire and that of wholly cold-rolled material. The pin-hole crystallograms of metal elongated by wire drawing are characterized by a six-fold symmetry in the bright spots of the rings whereas exclusively rolled material shows a four-fold symmetry. Figs. 11A, 11B and 11C show the three types as finished. It is interesting to note that heating for considerable time above the critical point and cooling (either slowly or by quenching) does not efface the preferred orientation, but does weaken it somewhat. This is shown in the three comparison crystallograms of Figs. 11D, 11E and 11F. This point will be referred to subsequently. Even four recrystallizations by allotropic transformation do not entirely remove the preferred orientation.

The quenched strip was treated in liquid air to remove all the retained austenite possible, but this process did not change the crystallograms visibly. The estimation of hardness of such thin strip presents a problem which was not entirely solved. It was brittle, of course, and tiny fragments of the material properly held would cut glass, but all attempts to obtain hardness by penetration methods failed. As tested for scratch-hardness it was classed

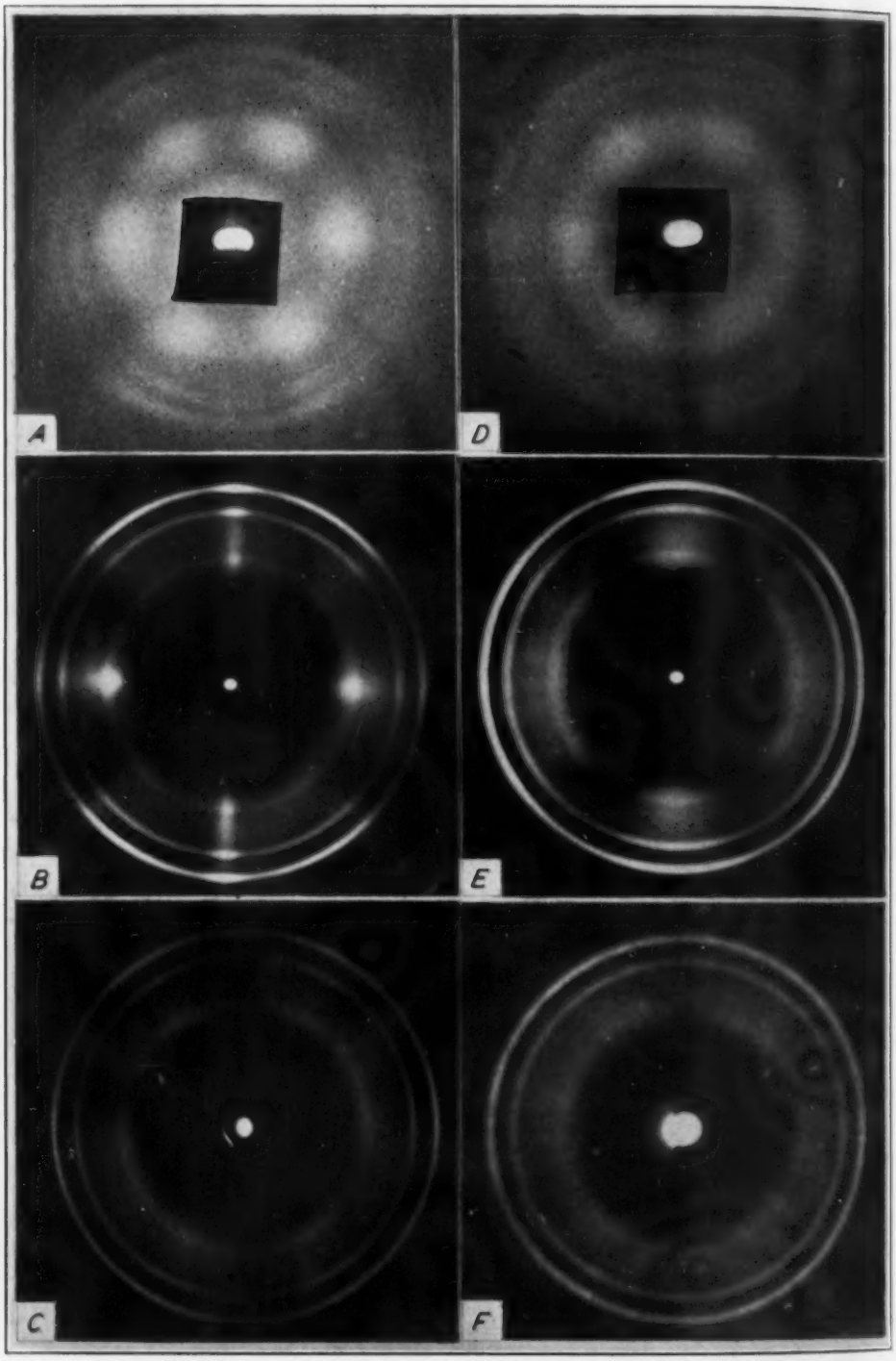


Fig. 11—Pin-hole Pictures Showing Preferred Orientation.
A—Developed by Wire Drawing.
B—Developed by Cold Rolling.
C—Developed by Both Processes.
D—Persisting after Heating A to 955 Degrees Cent. (1750 Degrees Fahr.) and Tempering 480 Degrees Cent. (895 Degrees Fahr.).
E—Persisting after Quenching B from 900 Degrees Cent. (1650 Degrees Fahr.).
F—Persisting after Quenching C from 900 Degrees Cent. (1650 Degrees Fahr.).
These crystallograms made from wide specimen; A and D are from the laboratory of Dr. Norton, courtesy of E. E. Legge, American Steel and Wire Co., Worcester, Mass.

merely as very hard. Probably the most convincing, even though qualitative, test was carried out by laying the strip on glass and scratching with various materials. The polished surface was scratched slightly by a razor blade, and not scratched by a knife blade of Rockwell C 58 hardness. Considering these various circumstances the hardness was probably in excess of Rockwell C 62. The strip possessed *traces* of plasticity which would ordinarily not be observed in martensite, which may have been due to the fact that it would be quite impossible to build up very high macroscopic stresses in such thin material. The original material was 0.003 inch (0.08 mm) in thickness, and the slight scale which was thrown off largely in the water quench reduced the thickness by only a few ten-thousandths of an inch.

As the X-rays become more and more nearly parallel the energy falls off very rapidly, and as in this case the total divergence was only 24 minutes of arc, the exposures were very long. But with widely divergent rays the error of measurement of crystalline imperfection is in general too great.

Results. A number of exposures of martensite were made for use in grain count experiments, both as quenched and as tempered. Typical of these is the series on the same specimen shown in Figs. 12, 13, 14 and 15. They represent respectively the martensite as treated in liquid air, as tempered at 150 degrees Cent. (300 degrees Fahr.), as tempered at 300 degrees Cent. (570 degrees Fahr.) and as tempered at 550 degrees Cent. (1020 degrees Fahr.). Fig. 16 shows the crystallogram from a still smaller specimen, with about the same original austenite grain size, as quenched.

At a glance it will be seen that the crystallograms are not those obtainable from a colloidal-grain-sized material. The amount of such material if present is not sufficient to make itself distinctly manifest. The major portion of the sample is clearly composed of large units in which the plane spacings are fairly uniform, but in which there is considerable variation in orientation. By a scrutiny of the pin-hole crystallograms (e. g., Fig. 12) the number of these units may be computed approximately as follows:

I, Number of images in one circle, observed to be <75 and >65

2θ, Angle of inclusion, $<10°$ and $>7°$

f, Number of contributing families is 6, Hence

$$\text{Number of units} = \frac{180I}{2\theta.f} \text{ or } > 196 \text{ and } < 320$$

The volume of the specimen was about 0.01 mm^3

The average size of martensite units is therefore <0.00005 and >0.00003 mm^3

This unit size corresponds to an average cube edge of about 0.037 or 0.031 mm.

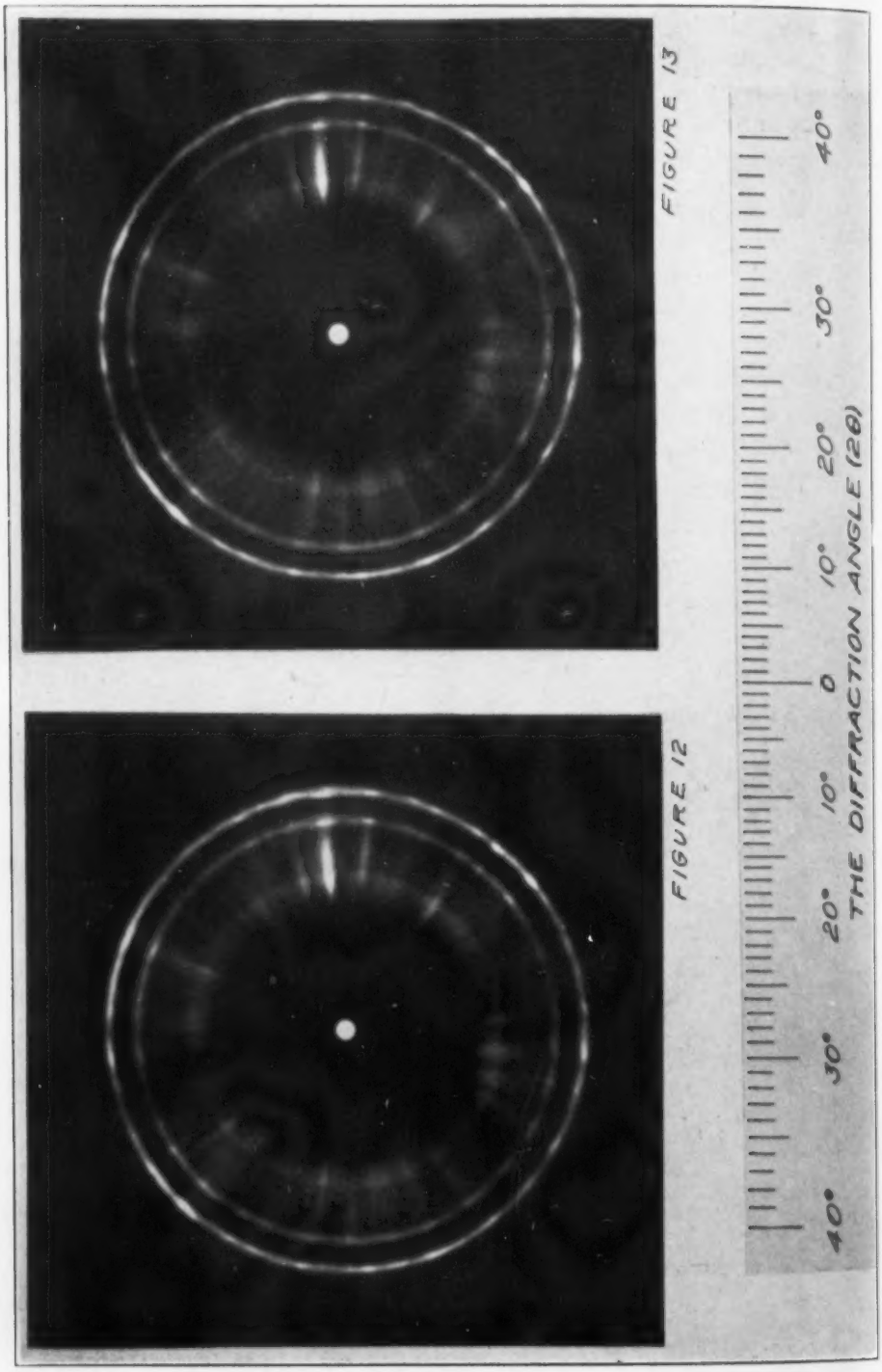


Fig. 12—Crystallogram of Strip Martensite as Quenched and Liquid Air Treated.
Fig. 13—Same as Fig. 12 Tempered at 150 Degrees Cent. (300 Degrees Fahr.)

From the photomicrographs of the martensitic strip the grain count of the original austenite may also be made. Choosing, for example, the photomicrograph of Fig. 7 it is found that >90 and <110 grains exist in an area of 0.09 mm². Again assuming cubic grains the average cube edge would be about 0.03 mm. This is in excellent agreement with the fact that there were always two or three austenite grains in the thickness of the specimen, 0.08 mm.

The number of crystalline units of martensite then, in given volume is essentially the same as the number of austenite grains prior to the quench. The agreement is far too close to be explained on any basis other than some influence of the one upon the other. It may be well to enquire into the nature of the distortion or imperfection of the crystalline units of martensite. All that can be said is that the units are continuous and have similarly spaced planes which may either be bent or twisted (Figs. 5C and 5D) through about 4 degrees of arc. The spacing is constant within about 3.5 per cent for the 1 1 0 planes, but is certainly much less constant for the planes of lesser spacing, indicating strongly the "solid solution type" of imperfection in a very exaggerated way. The actual degree of spacing variation in these 1 1 0 planes is less than that induced by severe cold work, but cold work does not influence the closely spaced planes as does the martensitic form of imperfection.

In order to determine whether or not the long radial images denoting bending or twisting could be produced by elastic deformation, mica plates, which, unstressed, produced very minute round images were elastically bent and exposed in the cameras. These plates were of a thickness comparable to the dimensions of an austenite grain. As bent, they produced the radial confusion found in martensite, but not to quite the same extent; the images were about two-thirds as long. Upon release of stress the images become undistorted again. Conceivably the distortion in martensite could be elastic. However, this is unlikely as will be shown.

Since the images of tempered martensite are very similar to those of martensite it is necessary to conclude that the bending or twisting undergoes only moderate change by the reheating. Clearly, a breaking up of a twisted grain into a very large number of platelets would not alter the image since it is already confused enough to blend such minute discontinuities. Indeed, martensite itself may, from X-ray evidence, be composed of platelets of minute dimensions,—perhaps up to about 0.001 mm in thickness, each twisted very slightly, perhaps through 0.1 degree from its neighbor. It should also be mentioned that very slight deformation applied to well-crystallized ferrite also

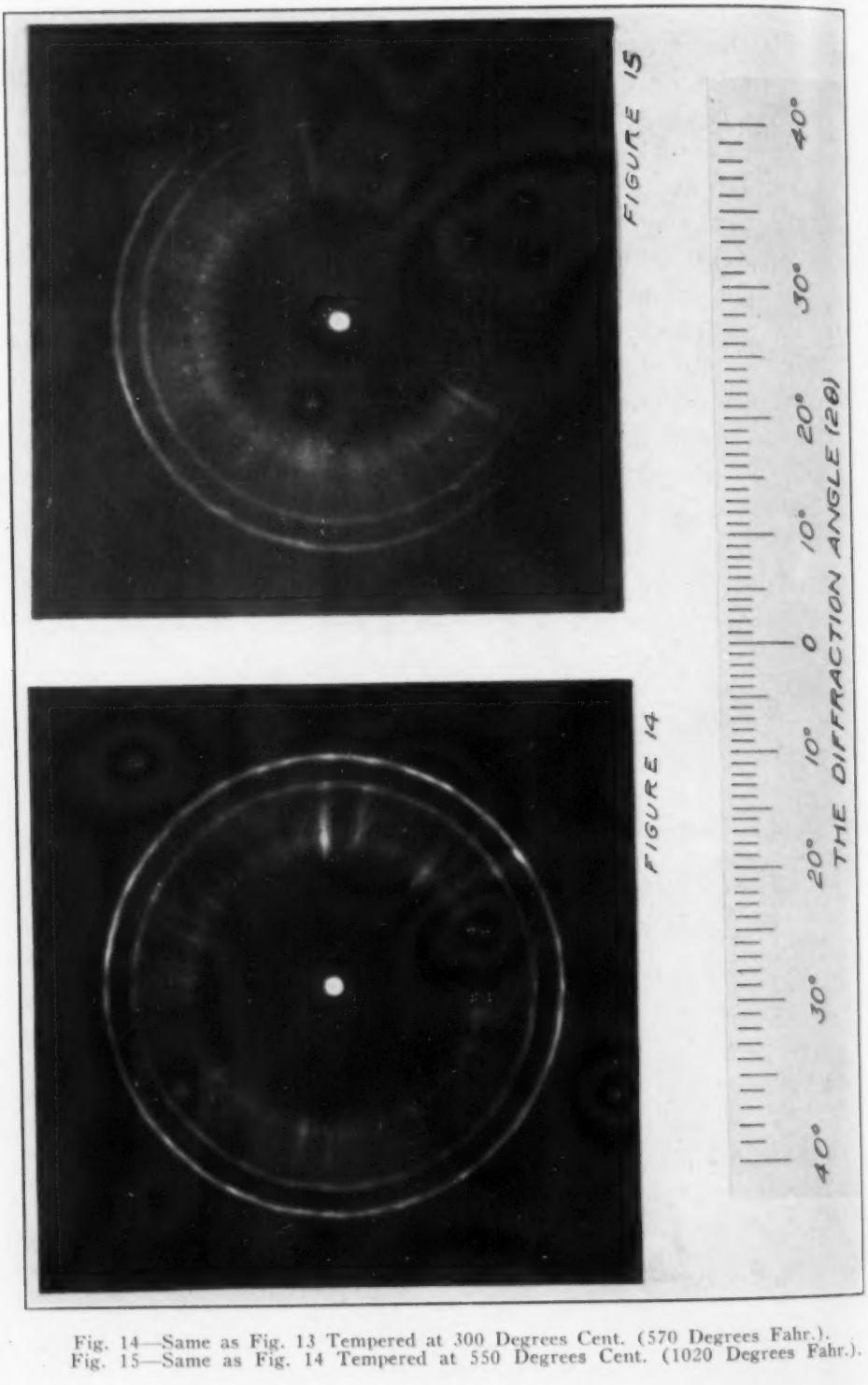


Fig. 14—Same as Fig. 13 Tempered at 300 Degrees Cent. (570 Degrees Fahr.).
Fig. 15—Same as Fig. 14 Tempered at 550 Degrees Cent. (1020 Degrees Fahr.).

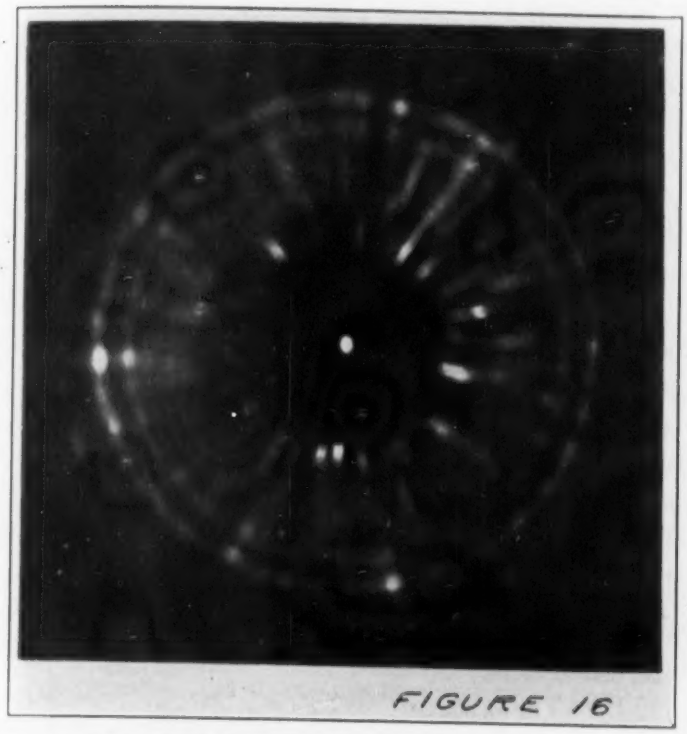


Fig. 16—Crystallogram of Massive Martensite with Extra Fine Pin-hole, 0.2 mm. Diameter.

produces the radial confusion in its crystallogram, Fig. 17, comparable, at least, with that found in martensite crystallograms. This indicates that the bent or twisted condition, per se, is not one contributing very generously toward hardness.

The brilliance of the monochromatic radiation as diffracted to the rings of the crystallogram indicates that a very considerable portion of the units of martensite are not bent or twisted beyond a fraction of a degree. This relative brilliance increases very slightly in the crystallograms of tempered martensite accompanying a narrowing of the total width of the monochromatic circles. In order to eliminate the radially elongated images in tempered martensite it is necessary to reheat the metal in the neighborhood of 600-650 degrees Cent. (1100-1200 degrees Fahr.), a temperature associated with considerable grain growth in moderately cold-worked iron and considerably higher than the temperature of first annealing of strain-hardened iron. It may be that the various platelets which result from the tempering of martensite (or perhaps existed in the martensite) coalesce and re-align themselves into *successively* larger layers of

perfect crystallinity with each successive increment of temperature or time, a temperature of 600 degrees Cent. (1100 degrees Fahr.) for many hours being necessary for the production of large ferrite grains of perfect crystallinity with no platelet condition remaining. Fig. 3 is a crystallogram of material held for several minutes at about 700 degrees Cent. (1290 degrees Fahr.). It represents the most perfectly crystallized ferrite grains in eutectoid steel that were obtainable under any of a number of conditions investigated. It will be seen that each image of the target in white radiation is nearly a perfect replica of the target distribution as shown in Fig. 6. It should be observed that with monochromatic radiation the crystals are too nearly perfect to image the entire target, but pick a limited portion only, actually two narrow zones, one for the molybdenum $K\alpha_1$, and the other for the molybdenum $K\alpha_2$. Such perfection of lattice is rare in metals.

Summary of X-ray Results. In the main the added refinements used in the X-ray study serve to establish the following items:

1. Martensite may be composed in the main of comparatively large "units" of ferrite.
2. The units have successive atom layers in some directions almost perfectly parallel, but successive layers are either bent (or more probably) twisted slightly on a "fibering" axis—only about 4 degrees per unit at a maximum. The fibering may be on more than one axis.
3. The units possess some systematic distortion besides the twisting (or bending) and have a marked random atomic irregularity of the "solid solution type."
4. Tempering does not remove the "uniaxial" perfection of crystallinity; nor does it restore the tri-axial perfection except it be at a temperature above about 600 degrees Cent. (1100 degrees Fahr.).
5. The size of the martensite units of ferrite is of the same order of magnitude as the size of the austenite grains from which the martensite was produced.
6. The orientation of the austenite grains controls the orientation of the martensite units, since preferred orientation persists after even four transformations.
7. A comparable degree of "twisting" is induced in carefully annealed steel with coarse grains and spheroidized carbides by slight cold work. The "twisting" (either continuous or

as thin platelets) does not induce hardness comparable to martensitic hardness.

8. In the "units" of martensite there are innumerable atomic planes of sufficiently large extent and with so little imperfection, that slip would occur at low stress in a ductile pure metal or *true* solid solution of like imperfection.
9. Solid solution of carbon (greatly supersaturated) in alpha iron remains as a possible or likely cause for practically the whole of the hardness of martensite.

IV. The Study With the Adapted Petrographic or Ore Microscope

(Through the courtesy of Dr. Olaf Andersen)

While metals are more nearly opaque to visible light than many substances yet thin sections, such as gold leaf, transmit light very perceptibly. Two years ago there were exhibited at the Werkstoff Schau in Berlin thin films of various metals which although free from porosity were yet sufficiently transparent to permit reading of print through seven such sheets at some distance from one to another and from the printed sheet. It is supposed that the property of "metallic lustre" is in some manner dependent upon this slight transparency. At any rate the effect is sufficient to permit the study of metals to some extent by means of polarized light.

The presence of relatively large crystalline individuals has been proved by examining the specimens in polarized light. This method is now used with considerable success in the study of opaque minerals, but does not seem to have been applied often to metallographic specimens. A brief description[6] of the method is therefore introduced.

For our investigations the ore microscope of E. Leitz, model MOP was used.

In the ordinary path of rays of the metallographic microscope are inserted two polarizing Nicol prisms, one in front of the vertical illuminator and one above it. The first Nicol, the polarizer, has its plane of vibration parallel or perpendicular to the axis of rotation of the reflecting mirror (the edge of the total reflecting prism) of the vertical illuminator. The second prism, the analyser, has its plane of vibration parallel to the main optical axis of the microscope and may be rotated around this axis. It is generally used with its

[6]The optical principles are treated on a mathematical basis by F. E. Wright; "Polarized Light in the Study of Ores and Metals," *Procedures*, American Philosophical Society, Vol. 58, 1919, pp. 401-447.

The essential points of the principles are discussed by M. Berek in H. Schneiderhöhn, "Anleitung zur Mikroskopischen Bestimmung und Untersuchen von Erzen und Aufbereitungsprodukten." Berlin, 1922. The same book also contains detailed descriptions of the technique of the method.

plane of vibration perpendicular to that of the polarizer (crossed Nicols). With this arrangement it is possible to distinguish between isotropic and anisotropic substances, and in the case of the latter to trace the outlines of crystal individuals.

An isotropic substance will remain dark in all positions when the specimen is rotated in the plane of its polished surface. The reason for this is that plane polarized light suffers no change in its state of polarization when reflected vertically from any plane surface of an isotropic substance. Thus the direction of vibration is unchanged and if the Nicols are crossed the reflected light will be extinguished by the analyser.

The plane polarized light reflected vertically by the surface of an anisotropic substance, on the other hand, will usually after the reflection have its state of polarization changed in several respects. Most important of these changes is the rotation of the plane of vibration. Due to this rotation the reflected light will have a component in the vibration direction of the analyser and some light will pass through. Thus, when the specimen is rotated in the plane of its polished surface, there is no permanent extinction between crossed Nicols as in the case of isotropic substances. Only in the positions in which the principal directions of vibration of the anisotropic surface coincide with the vibration directions of the Nicols will there be extinction because in these positions the rotation of the plane of polarization of the reflected light becomes zero. In other words an anisotropic substance between crossed Nicols becomes dark in four positions 90 degrees apart and light in all other positions. The color and intensity of the light are complex functions of several factors and cannot be discussed here.

Thus, in general, isometric crystals reflect polarized light essentially without change in intensity regardless of their position in reference to the crossed Nicols. Annealed ferrite and annealed austenite appear thus under the petrographic microscope. However, some of the non-cubic metals exhibit the characteristic alternate brilliant reflection and extinction as the specimen is rotated. In this class fortunately falls martensite, which as has already been discovered usually possesses a distinct departure from perfect cubic symmetry; this axial ratio in the tetragonal form is rather slight and, hence the effect is not based upon any very large fundamental atomic effect. In crystal aggregates of many fragments which in themselves may not be outlined by very definite grain boundaries the different fragments, by reason of their varying orientations, flash dark and light at different positions as the aggregate is revolved. Hence a method of determining grain-size and grain-shape is provided by the method. By the reflection method as applied to metals the contrasts are not nearly so brilliant as with translucent specimens which permit transmitted light.

The metal specimen might be a fractured surface, but the light reflection from such surfaces is very weak. The specimen is therefore polished to a plane



Fig. 18—Photomicrographs of High Carbon Martensite Quenched from 1200 Degrees Cent. (2190 Degrees Fahr.) into Water (Rockwell C64). × 700.
A—Ordinary Illumination.
B—With Crossed Nicols.
C—With Crossed Nicols, Specimen Turned 45 Degrees from B).

reflecting surface. But as polished there is no analyzing effect apparent. The polishing apparently develops such a random disturbance on the surface that the specimen reflects only a uniform intensity as the specimen is rotated. This uncharacteristic surface is removed by etching. It appears that etching is necessary to remove all such disturbed metal, but deep etching lessens the total light reflected very greatly, which at best is inconveniently low in intensity so far as photography is concerned.

The authors have not as yet succeeded in achieving a technique entirely satisfactory for this purpose, but improvement has been made and they hope ultimately to be able to produce photomicrographs of a quality comparable to the appearance in direct visual observation—and comparable with ordinary photomicrographs of metal.

Results. The two series of three photomicrographs, Figs. 18 and 19, illustrate the manner in which certain single portions of a field of uniform martensite appear under the conditions of the petrographic microscope. It will be noted that certain extended areas may appear almost black in one position and that at 45 degrees become highly reflecting. That the various units of martensite are anisotropic is of less consequence than the fact that the areas of comparatively uniform orientation are very large. In the specimens examined the units vary in size from an average diameter of 0.015 mm. to 0.06 mm. in high carbon steel (after prolonged heating prior to quench) and are as large as 0.15 mm. in low carbon hardened steel with an austenite grain very coarsely developed.

Some interesting evidence of imperfections are apparent in all specimens. The uniformly oriented areas are not always precisely identical with what would be interpreted as original austenite grain boundaries. The edges seem slightly differently oriented and are often ragged in outline. Occasionally the orientation of one unit will extend into an adjacent unit to a small extent. The most interesting observation is made that the "needles" as seen in the ordinary photomicrographs of martensite have little, if anything, to do with the structure with polarized light. Only one detail persists and that is the very frequent appearance of parallel lines spaced at between 0.0004 mm. and 0.002 mm. The dark constituent between the white in the narrow lines seems not to change with rotation and may be austenite. Some strength is given this hypothesis by the fact that the areas seem even more evenly white or black after liquid-air refrigeration.

Different regions within one unit may reach most nearly complete extinction or most brilliant reflection at a point as much as 10 degrees

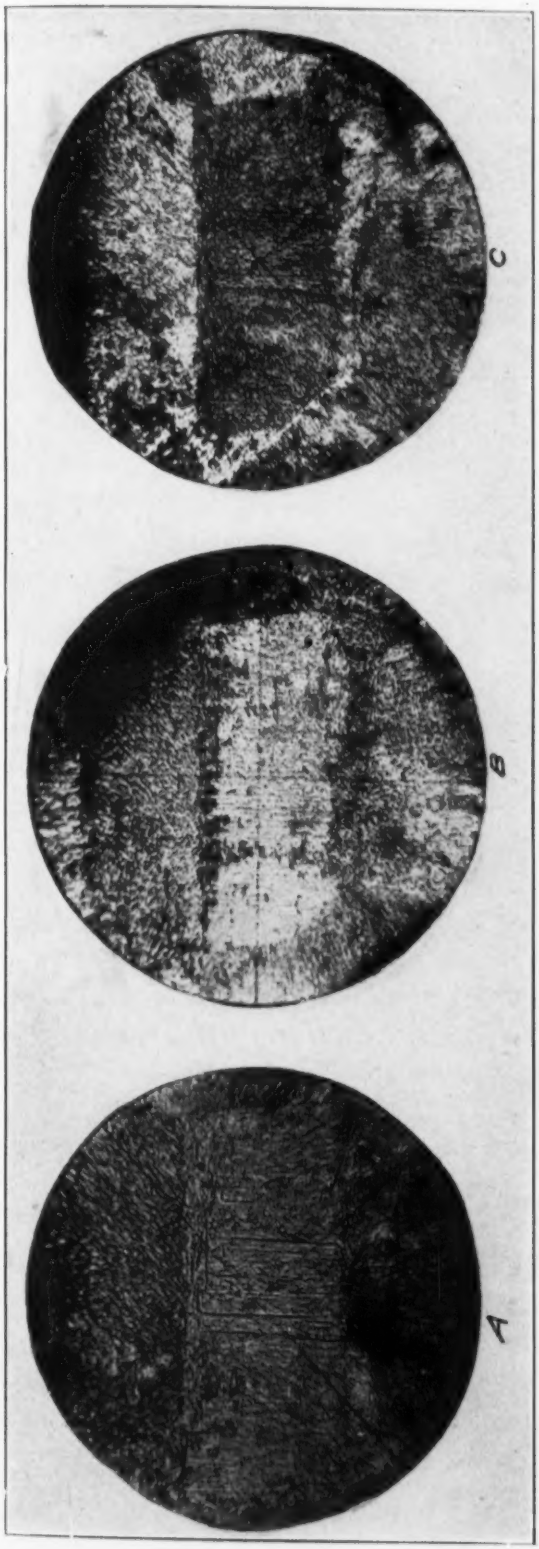


Fig. 19—Photomicrograph of Low Carbon Martensite Quenched from 1200 Degrees Cent. (2190 Degrees Fahr.) into Water (Rockwell 55C). × 280.
A—Ordinary Illumination.
B—With Crossed Nicols.
C—With Crossed Nicols, Specimen Turned 45 Degrees from B).

rotation from one another. Indeed in a few cases as the region under study began to flash into highest luminosity the light seemed to "sweep" across the area with the gradual rotation.

It perhaps is pertinent to mention that there is another circumstance in the deformation of martensite which points to the extended continuity in its crystal units. In the vicinity of Rockwell indentations, slight as they are, may be found a few definite slip bands in considerable variety. They are, however, extremely difficult to perceive and still more difficult to photograph. Their paucity would perhaps correspond somewhat to the very slight deformation possible in martensite. The length of the traces of the slip bands observed was of the order of 0.01 mm. or 0.005 mm. There are therefore reasonably long paths upon which the ordinary atomic-plane slip may occur in martensite as in the soft metals and solid solutions. The slip evidently occurs in such a limited way because of the great cohesion of the material and because rupture ensues after such limited deformation.

V. Hypotheses

The present investigation has produced results which are in the main in excellent qualitative agreement with the data of Gladding and Chartkoff[7] as reported. The quantitative nature of the present determinations makes possible a further consideration of some of the data offered. Within the last year the allotropic change has been observed occurring under the microscope.[8] The number of continuous martensitic units is about that of the austenite grains from which it originated. Even if the present estimations were in error 200 per cent the orientations would still be accounted for by the twins in the austenite if there be any, which seems probable.

Summing up the evidence it seems extremely probable that austenite decomposes to martensite by the slight simple movement of the atoms on its 1 1 1 planes. This process involves merely the change of the atom positions from a centered rectangle 2.546 x 4.42 (Ångstrom units) in austenite to similar rectangles about 2.860 x 4.047 in martensite. The 1 1 1 austenite planes resemble the 1 1 0 planes of fer-

[7] Zay Jeffries, "A Contribution to the Theory of Hardening and the Constitution of Steel." Campbell Memorial Lecture. TRANSACTIONS, American Society for Steel Treating. Vol. 13, 1928, p. 369.

[8] B. A. Rogers, "Change in Microstructure of Iron at A_3 Transformation Point." American Institute of Mining and Metallurgical Engineers. Technical Publication No. 218 (Iron and Steel No. 33) presented September 1929.

rite very closely indeed. The precise mechanism however is relatively unimportant. A grain of austenite yields so few orientations, according to the present study, as to permit several mechanisms for the process. Since the change from austenite to martensite involves a volume change the first martensite to form may be in compression and the martensite of the interior of a piece of metal may form under extensional stress (negative "ferrostatic pressure"). Such stresses can be computed to show in specific instances that the metal may locally be stressed beyond the ordinary elastic limit, but the absolute cohesion has probably not been approached, and in considering balanced microscopic stresses the ordinary figures for strength have no application.

Here we find great hardness in comparatively large units which in their *geometrical* structure offer no cause for hardness other than either an elastic twisting of about 4 degrees of arc per unit of 0.03 mm., or else a system of nearly parallel platelets very gradually differing in orientation from one another. When ordinary metals or solid solutions are given corresponding deformations they are not at all hard by comparison. While the theory offering grain size refinement as a considerable cause for hardness does not mention the degree of fineness in any quantitative term nor give dimensions of the grains it may be presumed that dimensions in the colloid range are intended. No units of this size have made themselves manifest by X-ray or other study, and the elimination process has not been complete enough to assign the hardness to one theory.

Only when martensite is produced by quenching from very high temperatures does the tetragonal condition result. It may be suggested that the only difference between quenching eutectoid steel at say 750 degrees Cent. (1380 degrees Fahr.) and 1050 degrees (1920 degrees Fahr.) is in the austenite grain size and the magnitude of the extensional stresses within the specimen. The tetragonal condition is obliterated by heating briefly at 100-150 degrees Cent. (200-300 degrees Fahr.) which temperatures could not well be expected to accomplish more than release of the severest stresses. The hardness may be very slightly increased by such treatment, or it may remain almost the same, or it may be slightly reduced. Hence, since the degree of tetragonal dissymmetry is proportional to the quenching temperature, the idea suggests itself that it is a measure of the microscopic stresses.

It has been suggested[9] that even at room temperature carbon precipitates from martensite and increases its hardness. The basis of this hypothesis is a certain similarity between the shape of the hardness-time curves of the precipitation-hardening alloys and the hardness-time curves of aging martensite. This analogy is very marked but need not be more than a chance similarity.

Most alloys and metals, which show no precipitation hardness by heat treatment exhibit a delayed hardening effect after "over-strain" as noted by Sykes and Jeffries[9] in their discussion of the aging of martensite. It would seem to the present authors that this phenomenon of over-strain accounts entirely for the age-hardening of martensite without recourse to the otherwise unevidenced carbide precipitation. There is ample stress and resulting strain to cause even low carbon material to exhibit over-strain phenomena; with the high carbon material capable of transmitting loads many times greater, we should expect the over-strain effect. Furthermore, there is an unexplained gap between the maximum hardness after 5 hours at 100 degrees Cent. and the first visible carbide precipitation effect over one hundred degrees higher. With all other precipitation-hardening alloys the maximum precipitation hardness results just as the visible precipitation effect upon etching is evidenced.

The authors therefore suggest that martensite grains (the units may as well be named grains, since the word has been previously applied to vaguely limited units) are greatly stressed by their formation and that the carbon is atomically dispersed. Since the degree of local and random atomic staggering in these grains is unusual the solid solution too may be somewhat unusual. The idea of pure position (geometry) explaining hardness, which is a matter of forces, has not been entirely satisfactory. The carbon atoms in addition to existing interstitially and thereby possibly greatly warping the ferrite lattice may also, as well, have nearly the full attraction or bonding to the adjacent iron atoms demanded by the compound Fe_3C. This speculation is based upon the strong (among intermetallic compounds) release of energy when Fe_3C forms. The ordinary solid solution between similar elements is, in terms of interatomic bonding, a very different substance from a supersaturated solution of carbon in alpha iron. The exact solubility of carbon in alpha iron at room temperature is, of course, not exactly known, being always super-

[9]W. P. Sykes and Zay Jeffries, "On the Constitution and Properties of Hardened Steel." TRANSACTIONS, American Society for Steel Treating, Vol. 12, 1917, p. 871.

saturated if carbide is visible, but eutectoid steel martensite must be supersaturated by about *25 times* its solubility for carbon. This condition is itself utterly unique.

The authors find ample substantiation of Jeffries' statement[7]. "The problem now appears to be one of appraising the contribution of solid solution and of grain refinement hardening. That both are hardening factors is generally admitted. That the two factors should cooperate to produce high hardness also seems logical." They find, however, no evidence requiring the existence of appropriately "fine" crystal entities of suitable arrangement to offer great hardness in martensite by means of the undefined grain refinement *alone*.

It remains yet to explain the peculiar hardness found in Rawdon's extraordinary quenched iron produced by arcing, and the hardness said to exist in Wyckoff's pressed iron powder. The arcing may possibly have fused the iron and then it may have frozen so rapidly as to form essentially amorphous iron (which should be hard). The examination of such hardened iron by expert microscopists and by means of X-rays should be helpful in determining its nature. At any rate, such phenomena seem to be utterly different from that of martensite formation by the customary processes which will not materially harden carbon-free iron even in their most drastic forms.

DISCUSSION

Written Discussion: By H. H. Lester, research physicist, Watertown Arsenal, Watertown, Mass.

Mr. Bain's and Dr. Heindlhofer's paper is of peculiar interest to me. It is somewhat revolutionary in that most of us have never considered it possible that martensite could be large-grained. However, their X-ray evidence seems to be sufficient and is quite convincing. It may be recalled that Mr. Bain was one of the pioneers in the X-ray study of martensite. He showed me the first X-ray film of steel that I ever saw. I believe he was first to discover that the Hull diffraction pattern for martensite consists of diffuse lines belonging to the alpha iron crystal spectrum. His results were, I think, used by Jeffries and Archer as a part of the foundation for their slip interference theory of hardness. Most of the scientists who have studied this subject have regarded the diffuseness of the spectrum lines as contributory evidence for the idea that the crystals of martensite are very small. We are now asked to revise this idea.

This diffuseness does not necessarily mean small grains, it may mean distorted crystals. Several years ago I made some studies of martensite and found, as did Mr. Bain, that the lines were diffuse. Further, I found the lines to be displaced from their normal positions. The measurements were

checked carefully, and seemed to indicate enlarged crystal patterns. On tempering at successively higher temperatures the lines became progressively less diffuse and approached progressively their normal positions, that is the positions they would occupy for pure iron. The lines became sharply defined and reached their final places only after tempering to 600 degrees Cent. (1110 degrees Fahr.)

We found another fact, which I believe fits in with Mr. Bain's results, that is carbide lines appeared faintly at tempering temperatures of 250 degrees Cent. (480 degrees Fahr.) These lines grew in intensity for increases in tempering temperatures up to 500 or 550 degrees cent. (930 or 1020 degrees Fahr.).

The above study led to the conclusion that martensite represents a condition in which the alpha crystals are distorted. From general consideration it was believed probable that they are also very small. Our evidence on this latter point, however, was inconclusive.

Since that time we have found in a study of cold-worked gun steel that, evidently plastic deformation tends to precipitate carbon or carbides from the apparently ferritic portion of sorbite.

The above results seem to support the conclusions of Mr. Bain and of Dr. Heindlhofer with regard to crystal deformation and the role played by carbon. Their evidence for large grains is entirely new, and will no doubt stimulate corroborative tests in other laboratories. The authors are to be congratulated on an excellent and a very important piece of work.

Written Discussion: By Albert Sauveur, Gordon McKay Professor of Metallurgy and Metallography, Harvard University, Cambridge, Mass.

Discussions and speculations as to why steel hardens on rapid cooling from an elevated temperature may be easily traced back to the phlogiston age. Ever since they have multiplied and increased in vigor, touching at times upon acrimony. The questionnaire to which the authors refer, sent in 1926 to 26 metallurgists of international reputation brought out the fact that while there existed considerable difference of opinion among them as to the exact reason why steel hardens on quenching, the majority believed in the existence of a solid solution of carbon in alpha iron and were inclined to consider it as the chief cause of increased hardness. Jeffries and Archer, on the contrary, completely discarded the possibility of the hardness of quenched steel being due mainly to the presence of a supersaturated solid solution of carbon in alpha iron. They contended that it resulted from the formation of extremely small grains of alpha iron. They called their theory the "slip interference theory", although obviously such designation would apply as well to any other explanation of the hardening of steel. Following Jeffries and Archer, the alpha iron present in quenched steel does not need to contain any carbon at all in solid solution in order to be extremely hard. It suffices that it be present in grains of submicroscopic (colloidal?) size. This view, which swept aside the opinion of previous investigators, was presented with much skill and persuasiveness and was accepted by many, I believe, as affording at last a satisfactory answer to the hardening of steel. On closer scrutiny, it must be apparent, however, that the theory is highly hypothetical. We rightly demand evidences of the

existence of grains of submicroscopic size. The best the sponsors of the theory can do is to refer us to the blurred or diffused appearance of the alpha lines in the diffraction spectrum of quenched steel. Other investigators, however, have claimed, apparently on as good ground, that such diffusion may result from the distortion of the space lattice caused by the presence of carbon atoms or of precipitated particles of the carbide Fe_3C.

The investigations described in the present paper supply us with the exact information needed to decide this controversial point. In view of the profound knowledge of X-ray analysis possessed by the authors, of their great manipulative skill and of the convincingly accurate interpretation of their results, it will be a difficult task for others to maintain that in hardened steel grains of alpha iron are present in submicroscopic dimensions. I welcome the results of this investigation as singularly clarifying. I welcome the conclusion that the existence of a supersaturated solid solution of carbon in alpha iron is probably the chief cause of the hardness of quenched steel, not only because it harmonizes more closely with my present view, but also because it appears to be the most logical. It is the view always held by Le Chatelier.

As I have had occasion to state elsewhere, the martensitic condition represents, in my opinion, an aggregate of hard needles embedded in undecomposed or retained austenite. When the authors of this paper refer to a solid solution of carbon in alpha iron being present in hardened steel and which, following the accepted nomenclature, they call martensite, they have in mind, I believe, the hard needles of the martensitic aggregate. It is not my understanding that they question the presence also of retained austenite in quenched steel. As explained in the reference cited, I consider the hard (martensitic) needles to be of the nature of troostite and hardened steel to be an aggregate, therefore, of troostite and undecomposed austenite. While, in my opinion, the major part of the hardness must be ascribed to the presence of an excessively supersaturated solid solution of carbon in alpha iron, I also believe that the hardness of the retained austenite, the solvent in which is necessarily undercooled, the fineness of the structure, the acicular character of that structure and the presence of severe internal strains may be important contributory causes.

It seems evident from the authors' interpretation of their results that the crystalline structure of alpha iron present in martensitic steel is not exactly similar to that of ordinary alpha ferrite. Are we not then justified in considering two types of alpha iron? In hardened steel alpha iron is described as possessing "some characteristic imperfections, distortion or crystalline idiosyncrasy" as departing "from cubic symmetry represented by a tetragonal system...." For brevity of speech, if for no other reason, could we not designate as beta iron this form of alpha iron? I find some justification for this suggestion in the consideration that it may well represent a condition assumed by iron in passing from its gamma to its alpha form. Beta iron would then represent imperfectly or incompletely transformed gamma iron, an assumption which would not differ greatly from Osmond's original contention as to the nature of what he designated as beta iron. I cannot refrain from expressing the hope that the authors will sometime apply their skill to the determination of the space lattice of pure iron between the A_3 and A_2 points. There is little doubt of its being

body-centered, but will it be clearly cubic or tetragonal? Whether body-centered cubic alpha iron should be considered allotropically different from body-centered tetragonal alpha iron must depend upon our definitions of allotropy. The results obtained by Dr. Anderson in studying the structure of hardened steel by polarized light are extremely interesting and hold much promise of yielding further valuable information.

Written Discussion: By Marcus A. Grossmann, metallurgical engineer, Republic Steel Corporation, Massillon, Ohio.

In spite of a disinclination to use superlatives, the present writer is impelled to congratulate Messrs. Heindlhofer and Bain on a piece of brilliant and scholarly work.

The paper will serve to clarify many points that have hitherto been obscure, among them the inheritance of "grain size" in steels after heat treatment. It is a familiar fact that steels, especially tool steels, which exhibit a coarse-grained fracture after forging or rolling will exhibit a similarly coarse-grained fracture after annealing and again after hardening.

The observation of these facts led the writer to the view that the orientation of the austenite grains influenced the orientation of the resulting ferrite grains upon cooling, and further that, upon heating, the orientation of these ferrite grains in turn influenced the orientation of the new austenite grains. Otherwise, that is, if the new orientations were always entirely random, there would be no relation in grain size after even a single transformation, let alone two or four such transformations.

However, doubts as to the above view soon arose, and for two reasons. First, the examination of grain size in annealed high carbon steels offers so many difficulties that evidence along this line was not available. Second, it seemed quite possible that "impurities", such as oxides or nitrides, might be dissolved in austenite and be rejected to grain boundaries and cleavage planes on cooling, and might thus affect the fracture in the cold state without reference to the crystallographic arrangement of the ferrite or austenite. No evidence on this view was available.

Thus for many years the factors in grain size inheritance have been obscure. Now, however, there can be little doubt that the parent crystal determines the size and the orientation of the new crystal, and that it does it in the manner indicated.

Written Discussion: By Dr. R. H. Aborn, Dept. of Metallurgy, Harvard University, Cambridge, Mass.

From the point of view of scientific writing this paper has set a new standard of excellence in the beautiful logic of its presentation. The critical discussion of X-ray methods is unique and will provide a rare treat to every thorough investigator who makes use of this important tool.

From the viewpoint of the advance of scientific metallurgy the perennial problem as to whether the observed properties of martensite arise from crystal size, crystal distortion, or both, is once and for all solved and in a most convincingly quantitative manner.

Another unique feature of this paper is the combination of X-ray and polarizing microscopic methods, in which the certainty achieved by the one is made doubly certain by the other. One obvious result would seem to be

that the factors in the Jeffries-Archer slip interference theory as applied to the hardness of martensite must be completely re-evaluated.

From a large number of measurements on the elastic distortion of iron crystals in an aggregate subjected to severe cold work (in tension) (published in Army Ordnance, 1925-6) the writer is inclined to disagree with the statement that "the actual degree of spacing variation (3.5 per cent) in these (1 1 0) planes is less than that induced by cold work." The maximum spacing variations for the first five families of planes observed by the writer were well within 1 per cent, and as long as the crystal structure remains approximately cubic he doubts the possibility of as large a variation as 3.5 per cent under any set of conditions.

Referring to Fig. 5 in the paper and denoting the ratio of increase of weakening of monochromatic diffraction intensity with increasing diffraction angle Θ to Θ itself by W_a, W_b, etc., for the various types of lattice imperfections it is probable that

$$W_a > W_b = W_{c,d} > W \text{ colloidal grains}$$

while $E_a < E_b < E_{c,d}$ (and $E_{\text{colloidal grains}}$ is negligible) where E_a, E_b, etc., refer to radial extension of white radiation diffractions. Mark and Hengstenberg (Naturw. Vol. 17, p. 443) have recently obtained quantitative data on the ratio of W in the annealed to W in the cold-worked condition for several metals.

The abnormally high value of W in the Hull patterns of martensite is now clearly shown by the authors to be due to the inordinate irregularities in the alpha iron lattice resulting from the remarkable degree of supersaturation with carbon, while the extreme radial extension of individual diffractions in the pin-hole or modified Laue pattern arises from the comparatively coarse grains either bent or twisted (or more probably both).

Some two years ago in examining the martensitic areas of Metcalf test bars, varying in carbon content from 0.5 to 1.4 per cent, the writer found distinct indication of the existence of a comparatively large crystal size. Again more recently, in examining one of the electrolytic iron specimens quenched in iced mercury from 1000 degrees Cent. (1830 degrees Fahr.) having the martensitic markings described by Prof. Sauveur and Dr. Chou, in their paper presented before the A. I. M. E. in Feb. 1929, the X-ray evidence once more clearly indicated the same comparative coarseness of crystal size. Having no further evidence at that time, however, the writer could scarcely hazard any opinion on the inheritance of grain size in quenching. It was interesting, indeed, to note the great difference in degree of confusion between this pattern and one from the Metcalf test bar made under identical conditions, the high angle diffractions of the latter being so weak as to be practically invisible in contrast to the comparatively sharp diffractions of the former.

The writer suggests that in freshly formed martensite, in addition to a relatively small amount of residual austenite, there may be varying amounts (dependent on the conditions of quenching and carbon content) of the transitional tetragonal and the stable cubic forms of ferrite highly supersaturated as to carbon, which is in practically complete atomic dispersion, with a mini-

mum of molecular Fe_3C formation of the type suggested by the authors.

The mechanism of aging of martensite would then involve a distinct increase in lattice distortion (causing the observed increase in hardness) as a result of the further formation of Fe_3C molecules within the ferrite lattice. At the same time the unstable tetragonal ferrite units would gradually transform to stable cubic ferrite.

On tempering, or aging at temperatures considerably above 100 degrees Cent. incipient crystallites of Fe_3C would begin to form, giving the troostitic structure and with this process the hardness begins to decrease.

There would be, of course, some overlapping of processes in these regions, but this would only slightly modify the substantial sequence of events.

There has recently appeared a somewhat similar hypothesis advanced by Gayler and Preston (*Journal*, Institute of Metals, 1929) to account for the observed aging properties of quenched duralumin.

Oral Discussion

R. L. Dowdell[1]: I am sure that we all appreciate the paper that Mr. Bain and Dr. Heindlhofer have presented. It seems from what Dr. Lester has said and from what Mr. Bain and his collaborators have said that there seems to be excellent proof to the statements they have made. There is one point, however, to which I would like to call attention.

If a good high carbon or alloy steel having a coarse austenitic grain size is quenched from some high temperature, it will usually retain this coarse austenitic grain skeleton at room temperature. After polishing and etching we will see a sort of martensitic slip across the grains with usually just one or two slips on the original austenitic grain. That would mean to a large extent that the grain size of the original austenite was frequently the grain size of the martensite, calling this martensite a grain. Of course, everybody thinks of something different when grain size is mentioned, but the above explanation is one way to look at it. Mr. Bain mentioned that he heated this material to 950 degrees Cent. (1740 degrees Fahr.) for 46 hours and the result was a fairly coarse grain size. If quenched from a lower temperature close to the normal Ar_3 or Acm depending on the steel, we will have a large number of these slips in many directions. The question is, would Mr. Bain's conclusion be true if he had a fine grain size of martensite to begin with instead of a coarse size. However, it appears that the method used by the authors is a very good one for measuring grain size.

Some time ago Dr. Harder and I made some determinations. We took steels of different carbon contents and cold-worked and quenched them from different temperatures. We determined their spectrograms, and in most cases we found the same conditions as Dr. Lester found, while in other cases we found a blurring of the lines. The cold-worked specimens looked just like the pieces that were water-quenched, and if we tempered them, the lines would usually form doublets. When we measured the centers of these lines, and calculated the lattice parameter we found points where the values would not

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agree at all with the different heat treatments, of hardening or tempering. There was no apparent relationship. Dr. Lester said that there was quite a definite relation. We did not find that. On taking cold-worked specimens and determining their lattice parameters you will generally find a large spread in lattice parameters according to different carbon content with no particular arrangement.

There is another point I would like to bring up. We wanted to see whether martensite had a fine grain size or whether it had a lot of strain to produce this blurred appearance. We produced gold and platinum colloids by arcing under alcohol, put them on the ultramicroscope and counted the particles which varied in size from 25 to 100 millimeters. Then we evaporated and measured them, and found the same blurry lines. We were then at sea as to whether this blurring was due to the fine grain or was due to lattice strain. One cannot solve the question with the Hull method so it looks like the pin hole method is highly desirable.

H. H. LESTER: In one case Mr. Bain showed the specimen which had been broken and examined close to the point of rupture. We did the same thing with the same thickness of material and found that after the rupture the lines were displaced sometimes to the right and sometimes to the left of the normal positions indicating that after severe plastic deformations the crystals are left, some in compression and some in tension.

DR. O. E. HARDER[2]: I think that Dr. Dowdell has covered most of the points which we should mention in discussing this paper. We have had in preparation a manuscript along this line for the past two years, but it will not work out. We rewrote the paper 2 or 3 times, and when we would get down to some of the things that we wanted to establish, whether we were removing strains or whether we were increasing grain size, it just would not come through. Mr. Bain and his associate have made a contribution which suggests that their method of attack is better than the method we used. I am very favorably inclined to their argument in regard to the grain size. It is only fair to say that from thinking of the possible mechanism involved in the formation of the martensite grain that I had reached a different conclusion than that which they have reached. They have established some very good evidence in that regard so that I shall probably have to revise my picture of the mechanism of martensite grain formation.

A. B. KINZEL[3]: Mr. Bain and his colleague, Dr. Heindlhofer, have certainly presented a very beautiful piece of work. It seems almost unnecessary to compliment them on it.

I wonder if Mr. Bain would care to say a word as to the evidence presented in the qualitative study made by Jeffries and Sykes presented before us about a year and a half ago in which the conclusions in certain respects were at some variance with those which he has presented here.

Authors' Closure

We are greatly indebted to all those who have contributed to this dis-

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cussion and sincerely appreciate the interest they have shown in this presentation. Their comment and criticism is the more welcome since some of the hypotheses are quite possibly controversial in character.

Dr. Lester is very kind in mentioning the early work of the authors, and it would be unfair not to mention that, in all probability Dr. Arne Westgren in Sweden observed the diffuse pattern of martensite before either of the present authors. However, independently, Dr. Jeffries and one of the authors had made such observations prior to the availability of the published work of Westgren in this country.

It must be admitted that the authors find some comfort in realizing that Dr. Lester, too, originally assumed that fineness of grain size (rather than characteristic imperfections of crystallinity) was the dominant cause of the diffuseness of the Hull pattern bands. The difference in pattern between these two states is, of course, not great and other methods would surely have to be employed to distinguish between the two types of diffuseness in X-ray patterns. And it is equally significant that Dr. Lester has more recently held that the X-ray evidence from the Hull method could as well be interpreted as indicative of distortion as of an exceedingly fine grain size.

Dr. Dowdell makes the very appropriate inquiry as to what the state of affairs would be in case the austenite grains in themselves were quite fine before transformation. We believe that the general case is in no wise modified. The resulting martensite would still have its orientation established by these smaller austenite grains, and the martensite units would be again approximately of the same magnitude as the parent austenite. One difference might exist. Since the actual linear change of dimension would be smaller in the transformation of a small grain of austenite, there would be smaller local stresses set up and less tendency to crack would result, as is well known to be the fact. The many fine grains could more completely compensate the distortions in a given volume and thereby reduce the maximum stresses.

Referring to the colloid experiments of Dr. Dowdell, would suggest heating the "protected" gold colloid to a temperature which would remove most of any cold work effect in case it existed. If the patterns made from the annealed powder were then still diffuse it would indicate that the single particles were of a size ranging between say 10 and 100 atom layers. Otherwise the patterns, if sharp, would indicate that the metal was originally stressed as produced by arcing under alcohol. In any such investigations there are certain considerations to be remembered; size of specimen, size of target, and the effect of these both upon single images from a single crystal and upon the Hull band of many images.

When Dr. Harder so generously mentions that the present paper may entail a revision of his concept of martensite formation, he also expresses very well the feelings of the authors when they were faced with the necessity of revising their views to conform with this evidence which seems to them fairly conclusive. Some of the theories of martensite which have been offered have been so simple and inherently attractive to the mind that it seemed they *must* be correct. Metallic behaviors are not limited to simple mechanisms.

Mr. Kinzel very appropriately inquires if the authors would comment upon

the study of martensite by Jeffries and Sykes as reported in the Campbell Memorial Lecture in Detroit (1927). This very important contribution, of course, has been considered very carefully by the present authors. Indeed the present paper attempts to answer the question implied in Jeffries' preface: "After concluding that the hardness of martensite can be attributed to grain refinement and carbon in solid solution, some researches are suggested which should help in estimating to what extent each of these factors contribute to the hardness of freshly formed martensite." Jeffries clearly intends to imply that grain refinement is the dominant factor whereas our findings give it little or no importance since we do not find that the grain is materially refined in martensite. Therefore we cannot exactly subscribe to the statement—"In the hardest steels produced recently by Sykes and the author it would appear that the hardening is due largely to grain refinement and the presence of iron carbide in very small particles. The reason for definitely assigning a minor place to solid solution in these steels is that they are apparently aged until most of the carbon is precipitated," unless "these steels produced by Sykes and the author" are in some way different from the general class. The reasons are: lack of any evidence for substantial amount of fine grains either in Gladding's martensite patterns or our own, and lack of evidence of precipitated carbide in any quenched steel heated short of the point for incipient troostite formation. We regard Jeffries explanation for the age hardening of the low carbon quenched iron as adequately explaining age hardening in quenched steel as well. This explanation is really the phenomenon of over-strain—in this case the "over-strain" was caused by the quenching stresses. An interesting explanation of this phenomenon is offered by Grossmann and Snyder, insofar, as cold work by mechanical means is concerned.

Furthermore, it will be recalled that in the precipitation hardening alloys (for example, Corson's hardenable copper alloys or Sykes' iron-tungsten alloys) a characteristic dark etching behavior accompanies the reheating for precipitation hardness. At the maximum hardness the polished section just begins to etch in a manner frequently likened to troostite. One has then, if he chooses to regard age-hardened martensite as containing precipitated carbides, to explain the total absence of the characteristic troostitic etching behavior. Unless heated for a few hours at a very much higher temperature than 100 degrees Cent. (210 degrees Fahr.) no troostitic etching would result in steel and yet that treatment produces a maximum age hardness—a state resulting in dark etching in known true precipitation alloys. It seems doubtful that the chemical attack is unreliable for detecting precipitation.

With the main points in Jeffries' paper we are in full agreement. That Gladding produced pin-hole crystallograms from a very few grains of austenite which showed rather more images of the target than might be expected from our deductions is probably due to the X-rays functioning to produce some Laue images as well as the customary ones. At least one film suggests that the crystal was situated to produce four Laue images simultaneously—a condition which is very rare and which would not recur often enough to vitiate a statistical method. In any case, the main deduction to be made is not at variance.

One point, however, seems somewhat important. Jeffries suggests that the "upsetting" theory offered some six years ago by one of us is entirely untenable and offers *instead* a transformation mechanism whereby the (1 1 1) planes of austenite rearrange to form the (1 1 0) planes of martensite. If one will but sketch the figure used in illustrating this "upsetting" theory he will find that the (1 1 1) planes of austenite actually become the (1 1 0) planes of ferrite and that the two mechanisms are identical, both involving ultimately the same "upsetting." The method of description alone differs, and the authors gratefully acknowledge the superiority of the (1 1 1) to (1 1 0) conception for purposes of exposition but must insist that they are clearly identical.

The authors have read the discussion so kindly offered by Professor Sauveur with the greatest pleasure. With his usual logical approach to metallurgical problems he makes a nice distinction between evidence and analogy, and rightly demands the former. With his views on the requisites of a theory of hardening the authors find themselves in complete agreement.

As to the existence of austenite in most hardened steel there can be little doubt. The authors invariably find it present to the extent of a few per cent except in the lower carbons. Any alloying material so far investigated increases the amount retained. Cooling to liquid air temperature removes most of it in plain carbon steel but is not effective in some higher alloy steels. It is not believed, however, to be an essential accompaniment to martensite. We prefer to regard martensite as that portion of fully hardened steel which is *not* retained austenite or residual carbide. We believe that martensite differs from troostite in possessing no precipitated carbide crystallites. The age-hardened martensite is, in our opinion, in a condition bearing the same relation to the freshly quenched martensite that the aged cold-worked iron or steel bears to freshly worked metal, but still with no carbide crystallites.

That martensite as originally formed is often found to be slightly tetragonal is now certain. It might well be appropriate to designate this state by a special name. However, the arguments against this would probably be that it is only elastically held in the tetragonal form and when warmed to 100 degrees Cent. (210 degrees Fahr.) it softens thermally sufficiently to permit the gradual and, ultimately more or less perfect restoration of the cubic condition, without substantial loss of hardness at room temperature. In all probability the long axis is so maintained by balanced stresses offered by adjacent grains whose minor axes are so maintained by compression. It must be borne in mind that in such martensite all axial ratios are present simultaneously; the ratio 1.06 is the maximum so far discovered, and that in addition the martensite cubic or tetragonal possesses the other distortions, both bending or twisting and atomic staggering. Possibly 1.06 is the absolute length to which unit length of the iron-carbon solid solution may be elastically deformed. This figure would refer only to fundamental crystalline characteristics represented by completely supported grains and not to actual tensile specimens which break long before the ultimate cohesion of the atoms is realized.

Mr. Grossmann makes a very pertinent observation upon the fact that coarse austenite yields a martensite whose fracture generally appears appropri-

ately coarse. Some more or less elaborate explanations have been offered to explain a "dry" or "sandy" fracture in "fine-grained" martensite. A coarse, hardened tool steel under the binocular microscope presents at moderate magnification the appearance of quite normal cleavage plane "facets." Mr. Grossmann pointed out this circumstance to one of the authors privately a number of years ago, and the fact of this apparent grain inheritance in steel free from burning or other abnormality might well have been offered as evidence favoring our present hypothesis. We do not, however, maintain that the austenite grain size is preserved during transformation when the product of the transformation is pearlite. Indeed there is evidence that the ferrite units of distinct orientation may be very fine indeed in fine pearlite.

The authors are grateful to Dr. Aborn for pointing out a mis-statement as well as for his very complimentary remarks. It is a welcome circumstance that Dr. Aborn has independently arrived at conclusions so closely checking those presented. Dr. Aborn's work on parameter determination is of the most accurate type. The statement should have been made that the total imperfections (including bending but excluding atomic "staggering") in cold-worked iron may exceed that of martensite. His hypothesis of a pre-precipitation condition whose hardness may be greater than any condition of particle precipitation is interesting.

In conclusion, the authors venture to offer for the consideration of those interested in these more or less theoretical aspects of metallography a thought on a possible condition of the carbon in martensite. In such an unusually supersaturated solid solution may it not be that the solid solution is itself somewhat different from the ordinary solid solutions of more similar elements? It seems possible that the carbon atoms are not substituting at lattice points but have already established a strong bonding with adjacent iron atoms to form, locally, un-crystallized molecules of Fe_3C.

Furthermore, there is already some evidence that alloys of the type of duralumin, Tempaloy, Sykes' iron-tungsten, lead-antimony, etc., are not at their maximum hardness at any point when or where fine particles are precipitated. It appears more than possible that any so-called critical particle-size of crystallite offers less contribution to hardness and strength than this pre-precipitation condition. If correct, this means that a true solid solution must first change (at any restricted area) to a condition such as described above for martensite wherein the solute atoms cease to function as substitute solvent lattice atoms, purely, and shift their interatomic forces to a different, and more stable distribution. Obviously, no solid solution can be produced of perfect homogeneity, and even the most nearly homogeneous ones may at some points pass to the softer condition of particle precipitation before the remainder of the alloy has lost its true solid solution characteristics and passed into the hardest pre-precipitation condition. The only "critical size" for maximum hardness would be the single molecule which would not constitute any particle precipitation whatever. To establish the facts concerning this suggestion will require much painstaking experimentation and the authors have made far too little progress to warrant more than the suggestion. However, private communications from other investigators give the idea at least a justification for its tentative suggestion.

THE ELECTROLYTIC DECOMPOSITION OF CEMENTITE AND AUSTENITE

By H. A. Schwartz, G. M. Guiler and H. H. Johnson

Abstract

For several years the author and others have sought to separate chemically iron carbide from ferrous alloys in order to study its properties. A year ago it was shown that the material isolated by the usual chemical methods might differ greatly in carbon content from the theoretical composition of the carbides and it becomes of interest to determine whether the residues separated differ in chemical composition from the carbides in situ.

The present paper deals with a more exhaustive study of the residue to determine its ultimate and to some extent, its proximate composition and is believed to show that carbides are very prone to decomposition when isolated by the methods in use in this laboratory.

It is shown that during the decomposition the residues take up hydrogen and oxygen most probably from the electrolyte and that the combined carbon which they contain differs in its chemical behavior from that of cementite.

Toward the close of the paper the composition of residues from austenite is discussed with a view to correlation with the decomposition products of cementite.

The value of the paper is believed to reside in placing on record the types of decomposition products which may be formed in order that other workers in the same field may not rashly conclude, as we did, that some of the more usual methods of isolating cementite leave that substance unattacked.

A year ago two of us with C. H. Junge presented data with regard to the chemical composition of the so-called carbides which may be separated electrolytically from white iron[1].

Largely in response to a suggestion by Hayes in discussion

[1]Schwartz, Johnson and Junge—"Graphitization of Prequenched White Cast Iron." Transactions, American Society for Steel Treating, Vol. 15, pp. 957-966.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. Of the authors, who are members of the society, H. A. Schwartz is manager of research, G. M. Guiler and H. H. Johnson, members of the research staff, National Malleable and Steel Castings Co., Cleveland. Manuscript received May 20, 1930.

of that paper that these compounds might not necessarily be pure carbides, certain chemical studies were undertaken whose results may have some interest as throwing light upon the character of the products which may form from cementite.

A metallographic study of the "carbides" in white iron has meanwhile been carried on by Bates and Lawson as an activity of the Metallurgical Department of Case School of Applied Science and will, it is hoped, be the subject of a separate publication. It is omitted here although relevant to the same subject.

The viewpoint of our earlier paper was that the carbon of our preparations existed either as graphite or as "combined" carbon in the sense of solution in or combination with iron.

Hayes suggested the possible presence of "graphitic acid" or other forms of carbon in combination with elements other than iron as a separate solid phase. Flotation on acetylene bromide (tetrabromoethane) was suggested as a method of separation dependent upon the difference in density of such compounds which might be great.

It was found that the carbides prepared electrolytically separated by such treatment into a heavier and a lighter fraction and that this took place whether the original preparation was high or low in carbon. The acteylene bromide apparently does not dissolve any iron in this separation, but if either fraction, and particularly the lighter, be subsequently washed with alcohol or ether to remove the reagent, iron passes into solution. An attempt to distill the reagent from the heavy fraction under diminished pressure resulted in a somewhat violent reaction apparently liberating bromine and forming ferric bromide.

It is possible that further experiment might have developed a more successful technique, but we felt that other more encouraging prospects for the solution of the problem existed. We suspended the sample in absolute methyl alcohol and separated the magnetic from the nonmagnetic substance by stirring with a suitable magnet. The magnetic fraction, except a small amount of apparently adherent material, was heavier than acetylene bromide and the nonmagnetic, except a few particles, was lighter than that compound. It seemed that the magnetic and flotation separations yielded identical results, the former being for us the more convenient.

Several years ago Edgar C. Bain, then with the National Lamp

Works at Cleveland, examined carbides of various compositions for us and considered their spectrograms indistinguishable from cementite; later, at the Union Carbide and Carbon Research Laboratories, he found the residue left on dissolving white iron in copper potassium chloride (graphitic acid?) to be so nearly amorphous as to yield no spectrogram at all. Our original preparations might, therefore, have been mixtures of cementite and "graphitic acid" which should have as the magnetic fraction, pure cementite.

We lack adequate opportunity for the precise determination of the Curie point or for measuring quantitatively the susceptibility of the magnetic fraction; however, a portion heated to 250 degrees Cent. (482 degrees Fahr.) in a glycerine bath was no longer attracted by an electromagnet. It is, therefore, apparent that the material has a Curie point in the neighborhood of that of cementite.

We analyzed separately the magnetic and nonmagnetic fractions of two of our preparations, one containing less than the normal carbon content of Fe_3C and one distinctly more, with the following results:

Table 1

Chemical Analyses of Specimens Tested

		No. 79	No. 75
Total Electrolytic Preparation.	Total Carbon	5.6	10.3
	Graphite	0.05	0.26
	Iron	81.0	64.4
	Silicon	1.40	3.55
	Manganese	0.34	0.30
	Sulphur	0.05	0.06
	Unaccounted for and error	11.61	21.39
	Nonmagnetic	44	81
	Magnetic	1000	1000
Nonmagnetic Fraction.	Total Carbon	1.40	10.8
	Iron (soluble in HCl)	57.9	36.5
	Silicon	1.69	4.5
	Hydrogen	0.38	1.50
	Chlorine	4.31	2.51
	Oxygen (excluding SiO_2)	31.00	39.00
	Error and undetermined	−1.58	+0.03
	Total Carbon	5.90	10.15
Magnetic Portion.	Graphite	nil	nil
	Carbon (Vol. in Cl in 20 min.)	2.69	3.69
	Iron (soluble in HCl)	80.75	67.30
	Iron (non vol. in Cl probably Fe_2O_3)	6.45	7.26
	Silicon (all as SiO_2)	1.12	2.51
	Hydrogen	0.31	0.20
	Chlorine	0.60	1.20
	Oxygen (excluding SiO_2)	9.86	15.00
	Error and undetermined	0.20	0.82

The determination of oxygen in this material may be somewhat open to question. We lack facilities for the vacuum fusion method. If substances of so high carbon content are treated by the Ledebuhr method, some of their oxygen is converted into CO while some carbon is converted into CH_4 making it impossible to judge from the CO_2 recovered from gas oxidized over iodine pentoxide how much oxygen escaped as CO. We therefore adopted the expedient used in the analysis of annealing house packings of taking the loss of weight on ignition to equilibrium in hydrogen deducting the observed loss of sulphur, the loss of carbon as obtained by analysis and the hydrogen content. The results come out quite concordant but may not perfectly represent the oxygen content. As a check, we have weighed the water actually formed and have invariably found the water to correspond to a major fraction of the oxygen reported, the balance being readily accounted for by the possible elimination of CO. To the oxygen so reported must be added that combined with silicon as SiO_2.

The carbon and iron content of the original mixture corresponds fairly well with that of the two fractions taken in their proper proportions which furnishes a check on the accuracy of the work. The low carbon preparation yields only about one-half as much of the nonmagnetic fraction as the other and this fraction is lower in carbon than the magnetic fraction. The two fractions of the higher carbon preparation are nearly equal in total carbon content and here, although we shall subsequently be interested in the nonmagnetic fraction, we find primarily a magnetic fraction far above the cementite carbon concentration which can not be a mixture of real cementite plus the nonmagnetic material, for the latter, if present, in some entrapped form, is not of sufficient carbon content to produce the observed results.

We are still confronted with the need of explaining the existence of a material much higher in carbon than cementite, magnetic at room temperature, nonmagnetic at 250 degrees Cent. (482 degrees Fahr.) and having an X-ray spectrogram not distinguishable from that of cementite. Except for its carbon content, we would call it cementite without the further investigation which disclosed the presence of large amounts of oxygen and other elements.

Can it be a mixture of cementite and another carbonaceous phase, the latter different from the nonmagnetic fraction both be-

cause of a very high carbon content and in being so completely enclosed in carbide as to be mechanically inseparable?

The analytical evidence precludes graphite as being the included contamination so that we are substantially where we started a year ago except that we now have a high carbon preparation demonstrably inseparable into two fractions by mechanical means, although the absence of minute inclusions high in carbon and completely soluble in nitric acid, has not been demonstrated.

Since the paper was prepared (May 20, 1930) there has appeared (*Zeitschrift für anorganische und allgemeine Chemie*, Vol. 190, page 145) an article by H. Thiele on "Graphite and Graphitic Acid" in which it is shown, among other things, that graphitic acid has the formula $C(OH)_3$, the carbon atoms have a hexagonal arrangement and that the substance is insoluble in concentrated nitric acid and easily decomposed by reducing agents, liberating its free carbon. The carbonaceous material resulting from the solution of white iron in the double chlorides being soluble in nitric acid is thus probably not graphitic acid although it might be a decomposition product thereof.

It will be noted that the O: H ratios of our preparation as shown in Table I, do not conform to the theoretical 16:1 of the formula nor is the C:O ratio the theoretical 3:2, the carbon being invariably much less. The insolubility of graphitic acid in concentrated nitric acid also suggests that its carbon would have been found with graphite in the experiment reported last year and in the present investigation.

When white iron (ferrite and cementite) is heated in dry oxygen-free chlorine, the iron volatilizes as Fe_2Cl_6. The method is one commonly used to determine separately the silicon present in the metal and as an inclusion containing SiO_2 and is described in various texts; for example, by Blair[2].

We find that, under these circumstances, and provided the time of chlorination is not more than enough to remove the iron, the carbon of the white iron, even when very finely divided, remains behind; quantitatively, repeated tests showing the same carbon content on a given metal, irrespective of whether the sample is burned in oxygen directly or after the volatilization of its iron in chlorine. The iron of cementite is completely converted into the chloride. If

[2]Blair—"The Chemical Analysis of Iron," page 68.

the time of reaction with chlorine is prolonged from the indispensable five to twenty minutes to say, fifteen hours, some 20 per cent of the carbon may disappear by volatilization in some form.

The residue from the solution of white iron in copper potassium chloride containing all the original carbon in a form other than graphite is practically completely volatile in chlorine in four or five hours and about equally so in nitrogen, which suggests that the removal is physical rather than chemical.

The carbon of the magnetic portion of either of the two preparations is nearly completely volatilized in chlorine in about fifteen hours, which would volatilize not over 20 per cent of the carbon of white cast iron and as shown in the table, the two volatilize 36 and 45.7 per cent of their total carbon respectively in twenty minutes.

We have thus a distincly different chemical behavior from that of cementite. We must not assume a mixture of either iron or cementite with some nonmetallic carbon compound, for the physical data preclude the existence of any large portion of alpha iron and the removal of carbon in chlorine by long treatment does not suggest a cementite origin for any part thereof. The ratio of iron to carbon not readily volatilized does not correspond to that of Fe_3C. Experiment has shown that the carbon remaining after volatilization of the iron by chlorine for twenty minutes is insoluble in 1.20 nitric acid. It is in this respect similar to that of cementite although differing in its reactivity with chlorine on long continued exposure.

As a check we chlorinated a sample of what appeared analytically to be a fairly pure cementite prepared by the electrolysis of wash metal, low in all elements except iron and carbon, but of unknown oxygen content, which was prepared in connection with earlier investigation by Mrs. Hird[3]. This preparation contained originally 6.3 per cent of total carbon and no graphite. After the usual short time chlorination, 4.5 per cent carbon was recovered.

This volatilization of carbon in chlorine, which does not take place from cementite bearing alloys, but does take place when the carbide phase is chemically separated, casts suspicion upon the identity of any chemically separated preparation, even if of unexceptional ultimate composition, with the carbide phase of a metallic alloy. It may be that our X-ray evidence is not applicable to cementite *in situ*.

[3]Schwartz and Hird—"Chemical Equilibria during Solidification and Cooling of White Cast Iron." *Mining and Metallurgy*, July 1924.

The behavior of the materials on heating is interesting. Portions of both fractions of each preparation were heated in a quartz tube at, roughly, 900 degrees Cent. (1652 degrees Fahr.) in a partial vacuum (exact pressure unknown but possibly around 1 centimeter of mercury). In every case there was a condensate in the cold part of the tube usually in the form of two dark rings representing apparently two degrees of volatility. On ignition in oxygen after removal of the boat and sample, carbon dioxide was evolved and a red stain remained suggesting the original sublimation of an iron compound subsequently oxidized to Fe_2O_3. The total loss on heating and the carbon in the sublimate each in terms of percentage of the sample heated, are tabulated below. The weight had reached what seemed to be a constant value.

Table II

Results Obtained On Heating Samples

	No. 79		No. 75	
	Magnetic	Nonmagnetic	Magnetic	Nonmagnetic
Total loss per cent	13.3	20.1	15.9	36.0
Carbon condensed per cent	0.6	1.0	1.6	0.55

The carbon of the nonmagnetic fraction of the low carbon preparation corresponds closely to the graphite content of the original metal. That of the high carbon preparation, however, is much greater, the graphite being sufficient to account for no more than 3.2 per cent carbon as against the observed 10.15.

We have examined also the residue from the electrolysis of an entirely austenitic manganese steel. The steel showed no metallographic evidence of cementite, had a permeability indistinguishable from that of air (within 20 per cent) and showed no alpha iron lines in the X-ray spectrum when examined for us by Norman P. Goss.

In this case the electrolysis was carried on in a closed cell using a mercury cathode and a very low current density of 0.00341 amperes per square centimeter. We believe that we have excluded all but the smallest traces of atmospheric oxygen from the electrolytic cell until the sample was thoroughly dry after washing with alcohol in a stream of hydrogen. The material so obtained was entirely nonmagnetic, pearl gray in color and lighter than tetrabromoethane and had the following composition:

Table III

Composition of Nonmagnetic Material

	No. 92	No. 93
Carbon	6.50	7.40
Graphite	trace	trace
Iron	11.00	8.60
SiO_2	53.03	61.9
Manganese	0.70	
Hydrogen	4.00	
Chlorine	1.10	
Oxygen	23.26	

The electrolytic gases were analyzed and found to contain carbon in some form similar to CH_4, i.e., not CO or CO_2. Only about one-half the carbon of the dissolved austenite was recovered in the cementite and of the remaining one-half, about one-third was accounted for by the electrolytic gas.

The remainder represents various losses by gas leakage, incomplete recovery of the residue and perhaps as water soluble organic compounds for the electrolyte after distillation from its iron content will bleach permanganate.

The iron content of the dissolved sample was almost perfectly accounted for by the residue and dissolved metal which suggests little loss of residue mechanically.

The sample listed as No. 93 in Table III was obtained from the same austenite by slow solution in 10 per cent hydrochloric acid with exclusion of air. Be it noted that these preparations take up oxygen from the electrolyte apparently by decomposing the water and independently of the use of current to hasten solution.

Conclusions

It has been shown that the so-called carbides isolated electrolytically from white iron consist of two fractions, a magnetic one of a density considerably greater than that of tetrabromoethane and a nonmagnetic one lighter than that reagent.

The magnetic fraction resembles cementite in having a Curie point and probably in atomic arrangement, but differs chemically in containing oxygen in considerable amounts and in the reactivity of its carbon to chlorine at 500 to 600 degrees Cent. (932 to 1112 degrees Fahr.).

The nonmagnetic fraction bears no similarity to cementite and

contains large amounts of oxygen. The carbon content of this fraction may, perhaps, be determined by the conditions of electrolysis which also possibly determine the relative amounts of the magnetic and nonmagnetic fractions.

The electrolytic or chemical decomposition of manganiferous austenite yields a compound similar in character to the nonmagnetic fraction from cementite though still more strongly oxidized. The oxidation is definitely by water and not by air.

Since the decomposition products of austenite are similar to those of cementite, it is possible that the carbon atom of austenite may have been chemically united to iron atoms although this is not necessarily so. Alternatively, one might consider that atomic carbon had combined with iron and oxygen as the material dissolved.

Hydrocarbons were observed to escape during the electrolysis of austenite representing either the decomposition of a "carbide" in austenite or the combination of "nascent" carbon and hydrogen atoms resulting from the reaction of acid on metal containing uncombined but dissolved carbon. The actual form of combination is unknown in any of our preparations.

It is interesting to note that some of the carbon and perhaps some iron of all of them is volatile in vacuo in a form which will recondense at room temperature.

Demonstration of the absence of oxidation is necessary before preparations of this character can be accepted as chemically identical with the carbide phase from which they were derived.

DISCUSSION

R. S. Archer:[1] I want to take the liberty of going into a little historical discussion which I think will interest all of us, and especially those of us who were acquainted with the late Professor Campbell.

Professor Campbell devoted much of his life to the study of the decomposition products of steel both hardened and hardened and tempered. Much of his work has received relatively little attention. I believe that perhaps he was out of touch of the investigative methods of the time because of his handicap of blindness. Perhaps for that reason he followed the channels of his early training in chemistry rather than the later methods of microscopy. Some of Professor Campbell's work was based on the old color method of determining combined carbon. He dissolved steels in nitric acid solution and hoped, by a study of the decomposition products, to arrive at a knowledge of the constitution of the steel be-

[1]Metallurgical laboratory, Aluminum Company of America, Cleveland.

fore solution. He found that the molecular weights of the products of decomposition increase with the tempering temperature of hardened steel. He interpreted this as meaning that the carbides in the steel reached a higher molecular weight by or association or polymerization.

In the light of our present conceptions it appears that much of this change which he called association of the carbide molecules is largely a matter of particle growth of precipitated carbides. When an austenitic or a freshly hardened steel is dissolved in nitric acid, much of the carbon goes into the production of compounds of low molecular weight. Some is lost in the form of gases. It was in the investigation of these gases that Professor Campbell lost his sight. As the tempering temperature goes up, the products which, in the case of freshly hardened steel impart a light yellow color to the solution, become darker in color. This seems to have a bearing on what Mr. Schwartz has told us in that it indicates that these organic derivatives can be formed from discrete particles of carbide because in the light of our present knowledge it seems quite certain that the bulk of the carbon is there in the form of precipitated carbides, at least in material which is tempered in the range to produce troostite. Professor Campbell published a paper, with M. B. Kennedy, in which he announced the discovery of a carbide corresponding to the formal Fe_2C. This, of course, corresponds with a higher carbon content than that of Fe_3C, and perhaps the paper by Mr. Schwartz and his associates explains that observation.

Leaving the discussion of Professor Campbell's work, it is of interest to note that the method of residue analysis as a means of studying alloy constitution has achieved a new dignity with the advent of the X-ray diffraction method of study. This method of residue analysis, that is, of the study of residue obtained by the chemical decomposition of alloys, has been thrown into considerable disrepute because of just the sort of thing which Mr. Schwartz has discussed; that is, that the residues obtained frequently do not correspond with the material which the investigators hoped to obtain and which they are studying. Frequently a check can be made as to the character of the residue by magnetic tests or other tests as Mr. Schwartz has outlined.

The X-ray diffraction method offers another excellent means of checking the residue. We have recently been employing this method with success in the study of aluminum alloys. From aluminum-titanium alloys we have separated a constituent, $TiAl_3$, and have separated it in such a degree of purity that the analysis checks almost exactly with the theoretical composition. The same has been done with $FeAl_3$. Some of this work is described in a paper at the Institute of Metals session on Thursday. In examining these residues, we first place them under a binocular microscope to see what we can learn by visual study. Frequently the particles may seem to have some particles of aluminum adhering to them. When this material is subjected to diffraction analysis, we can quite easily tell whether there is any appreciable amount of free aluminum there because if so, we get the characteristic lines with aluminum on the crystallogram. It is difficult to say what this method might have yielded in the case of

the material discussed by Mr. Schwartz, but any appreciable quantity of free metallic iron could probably be detected, and certainly if there was any appreciable quantity of a carbide other than cementite, lines would be obtained indicating the presence of such a different crystalline phase.

The point is that by the intelligent use of the diffraction method of study it is possible to check these residues and thereby obtain more benefit from a method of alloy constitution study which has not been greatly used in the past decade.

H. W. GILLETT:[2] It is quite possible that there may be some information on this subject which is not in print. Last spring I was talking to some of the people at the Bethlehem Steel Co., and they were somewhat perturbed about the question of cementite and other carbide residues in some of the inclusion work that they were doing. In discussing this problem the conclusion was reached that X-ray analysis offered an excellent method of chasing that matter down, and I believe Dr. Mehl is also following the matter up at the Naval Research Laboratory. I also know that work on the composition of the gases evolved from steel in different conditions of heat treatment, as a hoped-for method of finding what the constituents of the material are, is being prosecuted by Professor L. H. Rhodes at Cornell with the aid of one or two graduate students. That work has not yet gone very far but they felt that the initial work was encouraging. It might be worth while for those who are working along the same lines to get in touch with each other. Possibly even some of the same materials might be distributed so as to see how the different methods of attack work on the same material.

Authors' Closure

I have one very small item to contribute in closing. Very early in the game Mr. Bain (who was then located in Cleveland at General Electric) and I used to collaborate on that proposition. Not speaking as a direct authority, but merely speaking as a mouthpiece of what Mr. Bain told us, he was unable to distinguish these high carbon residues from the spectrograms of ordinary cementite. Those of you who may have seen the spectrograms will realize that the spectrograms of cementite are a combination sort of thing. I do not wish to commit Mr. Bain arbitrarily that there might not have been a few concealed lines in one that he overlooked in comparing, but they certainly were extremely similar, so much so that the ordinary man who only sees this occasionally would agree with Mr. Bain that they were line for line. One of the cementite spectrograms, if you picked certain lines out of it, would reproduce either alpha or gamma lattice, but there are a great number of those lines in the normal spectrogram. The cementite is either contaminated with iron or else you have it as part of the spacing of the cementite lattice and get it in that way.

[2]Director, Battelle Memorial Institute, Columbus, Ohio.

AUSTENITE DECOMPOSITION AND THE CHANGES IN MAGNETIC PROPERTIES*

By Harley S. VanVleet and Clair Upthegrove

Abstract

This paper presents a part of the experimental results obtained in a study of a 5-per cent tungsten magnet steel. The investigation had for its purpose the determination of a possible relationship between the magnetic properties and the austenite retained under different quenching conditions. Two methods of decomposing the retained austenite have been used, tempering and immersion in liquid air.

WHILE austenite, the stable form of the iron-carbon alloys above the critical temperature, is nonmagnetic in character, it has been suggested that possibly there is a critical amount of austenite, which for each composition of magnet steel yields a maximum permanence. If so, then it would be expected that the decomposition of austenite retained by rapid cooling was in some way related to the change in magnetic properties. Varying proportions of austenite may be retained at room temperature by the use of suitable quenching media when the quenching takes place from favorable temperatures. The austenite retained in the quenched condition may then be made to decompose during tempering at successively higher temperatures or during immersion in liquid air. It was the purpose of this investigation to study the effect of austenite decomposition in a five per cent tungsten steel on the change in magnetic properties. A relationship between the decomposition of austenite and the change in magnetic properties was anticipated.

Since it is well known that changes in the density and hardness of quenched steels occur during tempering, it was believed that by observing the changes in density and the changes in Rockwell C

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hardness that the range of temperature wherein austenite decomposition is most pronounced might be determined. Knowing the range of tempering temperature most favorable to austenite decomposition, then the changes in magnetic properties which occur within this temperature range could also be determined.

Using liquid air immersion as a means of decomposing the retained austenite, the change in density and changes in magnetic properties were also studied. Similarly by observing the changes in density of the quenched steel when subjected to immersion treatment in liquid air, it was possible to obtain the relationship between retained austenite and the change in magnetic properties.

Review of the Literature

During the past fifteen years, interest in the subject of retained austenite has steadily grown and many papers have been published relating to the preservation of austenite in quenched steels, and to its subsequent decomposition. In reviewing the work of previous investigators a brief outline of the conclusions pertaining to austenite is given. This is followed with a more detailed account of the work of a few investigators. The conclusions are outlined briefly as follows:

1. Austenite is shown by the method of X-ray spectrum analysis to be solid solution of carbon or iron-carbide in gamma iron.
2. Austenite is always present with martensite in medium or high carbon steels quenched from the normal hardening temperatures.
3. Higher quenching temperatures in general favor the retention of greater proportion of austenite.
4. The presence of certain alloying elements favors the retention of austenite at room temperatures in quenched steels.
5. Quenching in oil retains a larger proportion of austenite than either water or 5 per cent sodium hydroxide quenches.
6. Gamma iron and austenite are nonmagnetic in character.
7. Retained austenite shows a marked difference in stability upon immersing the quenched steels in liquid air. In the case of carbon steels liquid air immersion causes practically complete decomposition of retained austenite. In alloy steels no definite statement applies.
8. Austenite decomposition in liquid air causes an increase in hardness, expansion in volume, and the formation of martensite.
9. Austenite decomposition by tempering usually produces troostite.
10. When austenite is made to decompose on tempering, the temperature range most favorable for decomposition is dependent on the alloying elements present and their concentration. Time of heating at temperature plays an

important part in the decomposition of austenite during tempering. A long time at a low temperature will cause the same effect as a shorter time at a higher temperature.

11. In the tempering of quenched carbon steels, there are two processes which take place: first, the decomposition of martensite taking place over a wide range of temperature, beginning at a tempering temperature of 212 degrees Fahr. (100 degrees Cent.) and continuing until 752 degrees Fahr. (400 degrees Cent.) is reached; and second, the decomposition of austenite which takes place over a shorter interval of temperature, starting at approximately 302 degrees Fahr. (150 degrees Cent.) and ending before a tempering temperature of 572 degrees Fahr. (300 degrees Cent.) is reached.

12. Austenite retained in quenched steels is rendered less stable by deformation.

13. The hardness of the various metallographic constituents of hardened and tempered steel varies in order of decreasing hardness as follows: martensite, troostite, austenite, sorbite, pearlite, ferrite.

14. The density of the various constituents of steel are given in order of decreasing density, or increasing volume: carbides, austenite, ferrite plus carbide, and martensite.

The reader is referred to the work of Dowdell and Harder (1) for a more detailed account of previous investigations relating to retained austenite and its decomposition.

Nusbaum (2) showed the effect of tempering on the magnetic properties of a 1.04 per cent carbon steel quenched in oil and in water from 1472 degrees Fahr., (800 degrees Cent.). In tempering between 392 degrees Fahr. (200 degrees Cent.) and 572 degrees Fahr. (300 degrees Cent.) he obtained a decrease in hardness and coercive force Hc accompanied by an increase in maximum reduction Bm, and residual induction Br.

Mathews (3) showed the effect of liquid air immersion on the magnetic properties of a 5 per cent tungsten steel containing 0.74 per cent carbon. Upon liquid air immersion of the quenched steel, he observed an increase in the maximum induction Bm, residual induction Br, quotient Br/Hc, and Rockwell C hardness, and a decrease in the coercive force Hc. He points out the possible value of the quotient Br/Hc as a criterion for the quality of permanent magnets in preference to the product Br $\times$ Hc when comparing the results of different laboratories.

Dowdell and Harder (4) conducted liquid oxygen experiments on a number of austenitic steels. They conclude that the "austenitic structure produced in quenched steels have different stabilities and

that they show different amounts of decomposition when subjected to the liquid oxygen immersion. In the plain carbon steels, the decomposition goes practically to completion, while in the case of manganese and high speed steel there is little or no decomposition." They state that austenite decomposition in liquid oxygen appears to be a recrystallization phenomenon which shows itself first along the slip planes.

Bain and Waring (5) conducted liquid air immersion experiments on two steels: an oil hardening die steel containing 0.82 per cent carbon and 1.65 per cent manganese, and a stainless steel containing 0.30 per cent carbon and 12.78 per cent chromium. They concluded "that whatever changes are brought about by extremely low temperatures are not caused by a steep thermal gradient within the metal and the accompanying stresses. The uniform low temperature is apparently sufficient to destroy the austenite and further liquid air treatment accomplishes little or nothing. The increase in permeability and expansion indicates that austenite is removed, while the hardness increase and the other magnetic changes indicate the formation of martensite." The results of Bain and Waring show the effect of liquid air immersion upon the magnetic properties of the oil hardening die steel. They obtain an increase in hardness, an increase in length, an increase in maximum induction, an increase in residual inductance, and an increase in coercive force.

Honda (6) concludes that the effect described by Mathews, i. e., that more austenite is retained by oil quenching than in water quenching, is valid only for thick plates and rods. For thickness less than 2¾ millimeters Honda finds more austenite in the water-quenched specimens than in the oil-quenched specimens. When the thickness is greater than 2¾ millimeters, more austenite is retained by oil quenching. His experiments were concluded with a 2.96 per cent chromium steel containing 0.84 per cent carbon. Upon immersion of the quenched plates in liquid air the amount of transformation is less, the thicker the specimens.

Upon quenching from a high temperature into water, a series of carbon steels, varying in carbon content, Honda and Osawa (7) showed that more austenite was retained in the section nearer the surface than on the inner layer. This was affirmed by the relative intensity of the spectral lines, by microscopical examination, and by the distribution of hardness.

Tammann and Scheil (8) and Schroeter (9) conclude that the austenite to martensite reaction during immersion in liquid air actually takes place at the low temperature and not upon reheating to room temperature.

In a recent paper Maurer and Schroeter (10) estimated the austenite content of eight different quenched steels by means of the values of magnetic induction at the point of saturation. The percentage difference in magnetic induction of the steel in the quenched and annealed conditions was used as a criterion for the amount of austenite present. The following results were given for a steel containing 0.73 per cent carbon, 5.51 per cent tungsten, 0.41 per cent chromium and 0.18 per cent manganese.

Quenching Temperature Degrees Fahr.	Degrees Cent.	Quenching Medium	Austenite As Quenched	Immersion in Liquid Air
(1472)	800	oil	10.0	8.5
(1652)	900	oil	13.0	9.5
(1832)	1000	oil	12.5	9.5
(2012)	1100	oil	11.5	8.0
(2192)	1200	oil	11.5	7.0
(1472)	800	water	6.5	6.0
(1652)	900	water	8.5	7.5
(1832)	1000	water	8.5	8.0
(2012)	1100	water	8.5	7.0
(2192)	1200	water	8.0	...

Experimental Procedure

The experimental procedure falls naturally into three parts: first, choice of a material; second, procedure in heat treatment; and third, procedure in collecting experimental results.

Choice of a Material. The steel used for this investigation of the effect of the decomposition of retained austenite on magnetic properties was a tungsten magnet steel. The composition is given by the following chemical analysis:

	Per Cent
Carbon	0.60
Tungsten	5.70
Manganese	0.27
Vanadium	0.11
Chromium	0.36
Sulphur	0.007
Silicon	0.23
Phosphorous	0.014

This steel was used in preference to several other steels on account of the fact that it gave, in a few preliminary experiments, the greatest change in density upon quenching in comparison with the other steels available at the time this investigation was started. It was thought that the greater change in density upon quenching indicated more austenite retained at room temperature.

The steel was furnished in three-eighth inch round bars approximately thirty inches long. Bars to be used in magnetic determinations were cut to a length of ten inches, while the bars to be used in density and hardness measurements were cut into five-inch lengths. The five-inch bars weighed approximately sixty-five grams in air.

Heat-Treatment. Previous to the quenching treatment the various sets of bars were given a preliminary heat treatment by heating for a period of six hours at 1600 degrees Fahr. (871 degrees Cent.), followed by cooling in air. The normalizing treatment was carried out by suspending the bars in an electric tube furnace.

In order to have a convenient datum plane on which to base the changes in properties of the magnet steel, it was decided to use the properties existing after annealing for a period of four hours at 1750 degrees Fahr. (955 degrees Cent.) followed by slow cooling in the furnace. Three ten-inch bars and three five-inch bars were suspended in the same furnace used in normalizing.

In conducting the quenching treatments a standard practice was adhered to in order to eliminate as many variables as possible. The five-inch bars for density determinations and the ten-inch bars for magnetic measurements were quenched simultaneously. The bars were placed in the cold furnace, heated with the furnace to the quenching temperature, held for the predetermined time and quenched. Before the bars reached the temperature of the quenching medium, they were removed and placed in boiling water, where they were held for a period of one and one-half hours. This procedure was previously found to prevent quenching cracks at the higher quenching temperatures. In quenching the bars the descent into the quenching medium was made vertically and as rapidly as possible. The details of the quenching treatments are given in Table I. The time of soaking at the quenching temperature was made long in order to give the carbides and tungstides ample time to go into solution. Owing to the two hour heating period at the quenching temperature, the outer surfaces of the bars suffered some decarburization. The extent of this decarburization was checked by metallographic examination and was found not to exceed one one-hundredth of an inch in the bars quenched from 1900 degrees Fahr. (1038 degrees Cent.).

In a like manner the tempering treatments were carried out in accordance with a definite plan. The time at temperature was 120

minutes excepting at a temperature of 212 degrees Fahr. (100 degrees Cent.), where the time of tempering was twelve and one-half hours. The bars were tempered at 212 degrees Fahr. (100 degrees Cent.) in water, at 392 degrees Fahr. (200 degrees Cent.) and 572 degrees Fahr. (300 degrees Cent.) in oil, and at the higher temperatures in

Table I
Quenching Treatments

Quenching Temperature	Quenching Medium	Temperature of Quenching Medium	Time at Quenching Temperature in Minutes	Type of Heating
1600° F (871° C)	Oil	22° C	120	Electric tube furnace with charcoal.
	Water	21° C	120	
	5% NaOH	20° C	120	
1700° F (933° C)	Oil	20° C	120	Electric tube furnace with charcoal.
	Water	22° C	120	
	5% NaOH	23° C	120	
1800° F (983° C)	Oil	27° C	120	Electric tube furnace with charcoal.
	Water	32° C	120	
	5% NaOH	37° C	120	
1900° F (1038° C)	Oil	23° C	120	Gas fired muffle furnace with charcoal.
	Water	23° C	120	
	5% NaOH	19° C	120	
2000° F (1097° C)	Oil	26° C	90	Gas fired muffle furnace with charcoal.
	Water	26° C	90	
	5% NaOH	24° C	90	

an electric tube furnace. The bars to be tempered were placed in the cold furnace, heated to the desired temperatures, held for 120 minutes, removed from the furnace and allowed to cool in air. Successive tempering operations at higher temperatures were performed on the same set of bars.

Immersion treatment in liquid air was carried out in four periods: 8 hours, 52 hours, 58 hours, and 57 hours, making the total time of immersion equal to 175 hours. The bars were placed in a Dewar tube about 13 inches deep; liquid air was then poured carefully over the bars a little at a time in order not to cause excessive boiling. Additions of liquid air were made frequently to maintain the level above the ends of the bars. During the last 12 hours of the longer runs, no additional air was added and the liquid allowed to evaporate, although in no instance was it completely evaporated at the time of removing the bars from the tube.

Methods of Collecting Results. Density determinations, magnetic measurements, and Rockwell C hardness values were made on the tungsten steel bars for the various conditions of heat treatment

and periods of immersion in liquid air. The methods used in making these measurements will be discussed briefly in the following paragraphs.

The tungsten steel bars used in the determination of the density were carefully prepared by polishing with coarse and fine emery paper, followed by washing thoroughly in alcohol and ether. After washing, the bars were handled with a small piece of chamois skin in order to prevent corrosion from the moisture of the hands.

The density was determined by the Archimedean principle. The procedure was the same as that described by Norbury (11), in which special precautions were outlined to eliminate as far as possible the errors arising from surface tension.

The magnetic properties were determined with a direct current Fahy Simplex Permeameter. The values of residual inductance and coercive force were obtained by plotting the hysteresis curves for the several conditions of heat treatment and immersion treatments in liquid air.

The Rockwell C hardness values were taken with a diamond point penetrator under a total load of 160 kilograms. Specimens used in hardness measurements, having a length of approximately three-eighths of an inch, were cut from the ends of the density bars after each treatment. The specimens were prepared by polishing both ends of the small cylinders with number zero (No. 0) emery paper, insuring a uniform surface condition of all specimens. The average of five readings was taken as the hardness of the specimen.

Experimental Results

The discussion of the results obtained conveniently divides itself into two parts: first, the discussion of results obtained when the austenite is decomposed upon tempering; and second, the discussion of results obtained when the austenite is decomposed during immersion in liquid air.

Results of Tempering Experiments. Since the variation in density and hardness of quenched steels during tempering treatments are fairly well known, it was thought, by observing the changes in slope of the curves representing the change in density and the change in hardness, that the range of temperature most favorable to the decomposition of austenite might be determined. Knowing this temperature range, it would then be possible to determine from the curves representing the changes in the magnetic properties which occur upon

tempering, those changes which may be attributed to the decomposition of austenite. The data for the curves representing the changes in properties of the tungsten steel upon tempering at successively higher temperatures were calculated, using as a basis the values obtained in the annealed condition. The basic values are given in Table II for an anneal of four hours at 1750 degrees Fahr. (955 degrees Cent.).

Change in Density. In every instance, referring to Table II, the density of the oil-quenched specimens was greater than the density of the bars quenched in water or 5 per cent sodium hydroxide. This indicates a greater retention of austenite in the bars quenched in oil, which is in agreement with the view of Mathews and others. A greater retention of austenite in the bars quenched from the higher temperature is indicated by the greater change in density during the quenching treatment. In considering the water and 5 per cent sodium hydroxide quenching treatment no definite statement covers all instances. When the quenching temperature was 1800 degrees Fahr. (983 degrees Cent.) or above, the specimens quenched in water showed a greater density than those quenched in 5 per cent sodium hydroxide. On the other hand, when the quenching was carried out below 1800 degrees Fahr. the density of the bars quenched in 5 per cent sodium hydroxide gave the greater density.

The curves representing change in density with tempering temperature for the bars quenched from 2000 degrees Fahr. (1097 degrees Cent.) are shown in Fig. 1. Three distinct changes in the slope of the curves were found common for all quenching temperatures: first, at 572 degrees Fahr. (300 degrees Cent.); second, at 977 degrees Fahr. (525 degrees Cent.); and third, at 1121 degrees Fahr. (605 degrees Cent.). These changes in slope of the curves representing changes in density with tempering temperature may be attributed to the separate or combined influence of the following factors.

1. Release of quenching stress and distortion of the space lattice, resulting in an expansion in volume or a decrease in density.
2. Precipitation and coalescence of carbide particles resulting in a contraction in volume or an increase in density.
3. Decomposition of martensite causing a contraction in volume or an increase in density.
4. Decomposition of austenite causing an expansion in volume or a decrease in density.

In addition to the three common changes in slope for all quench-

Table II
Tabulated Experimental Findings

Quench Condition, Hmax = 200 Gilberts/cm

Quenching Temperature	Quenching Medium	Gauss Bm	Gauss Br	Gil/cm	Br × Hc	Br/Hc	Density of Bar for Liquid Air Immersion	Mean Density of Two Bars for Drawing Experiments	Rockwell "C" Hardness
1600° F (871° C)	Oil	12790	7970	47.1	375,000	169	8.0786	8.0787	64
	Water	14090	9410	47.0	442,000	200	8.0643	8.0647	63
	NaOH	14180	9340	46.8	437,000	199	8.0652	8.0655	62
1700° F (933° C)	Oil	13730	7930	44.3	351,000	179	8.0774	8.0775	62
	Water	14380	9320	44.5	415,000	209	8.0680	8.0672	62
	NaOH	14420	8800	44.5	391,500	198	8.0681	8.0690	62
1800° F (983° C)	Oil	12220	7420	47.1	349,000	158	8.0761	8.0762	62
	Water	12800	7970	45.1	359,000	177	8.0695	8.0696	63
	NaOH	13410	8280	43.3	359,000	191	8.0667	8.0666	62
1900° F (1038° C)	Oil	13300	8200	49.1	402,500	167	8.0706	8.0716	62
	Water	13850	9040	45.5	411,000	199	8.0647	8.0641	63
	NaOH	14300	9350	43.6	407,500	214	8.0614	8.0619	63
2000° F (1097° C)	Oil	13050	7700	44.5	342,700	173	8.0660	8.0664	62
	Water	13890	8280	39.8	329,500	208	8.0634	8.0624	63
	NaOH	13650	8490	41.9	355,700	203	8.0601	8.0600	64
Normalized at 1600° F (871° C) for 6 hours.		15540	9060	24.1	218,500	376	8.0933	.....	36
Annealed at 1750° F (954° C) for 4 hours.		16540	11650	10.7	124,700	1089	8.0977	.....	26

ing temperatures, the curves for the bars quenched from 2000 degrees Fahr. (1097 degrees Cent.) show a slight decrease in density upon tempering between 392 degrees Fahr. (200 degrees Cent.) and 572 degrees Fahr. (300 degrees Cent.). A similar effect was observed for the oil-quenched bars only, when the quenching temperature was

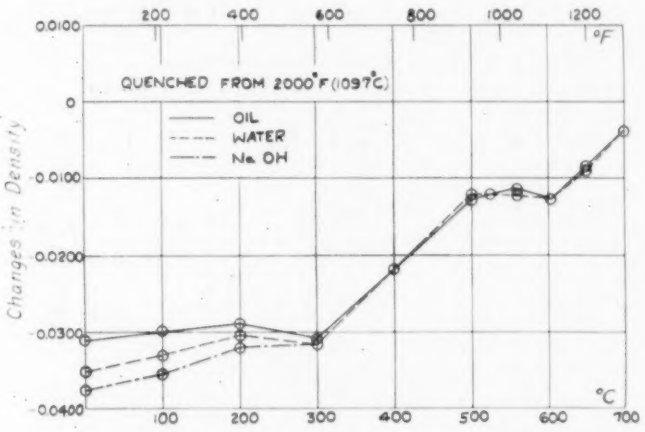


Fig. 1—Change in Density upon Tempering. Values Obtained in Annealed Condition Were Used as a Basis.

1900 degrees Fahr. (1038 degrees Cent.) and below. The amount of decrease was greater at the lower quenching temperature of 1600 degrees Fahr. (871 degrees Cent.). This effect is believed to be a result of a partial decomposition of austenite, fourth factor, upon the tempering of the oil-quenched specimens.

The increase in density which occurs upon tempering up to 392 degrees Fahr. (200 degrees Cent.) in the water-quenched bars and in 5 per cent sodium hydroxide quenched bars is probably a result of the predominant influence of the second factor, precipitation and coalescence of cementite particles. This conclusion is indicated by the relative increase in density between the oil, water and 5 per cent sodium hydroxide quenched specimens. The increase in density is least for oil where precipitation during quenching takes place over a longer period and greater where precipitation is retarded by more rapid quenching.

From Fig. 1 it is observed the differences in density between oil, water and 5 per cent sodium hydroxide quenched specimens disappear when the tempering temperature reaches 572 degrees Fahr. (300 degrees Cent.). This is believed to be a result of relieving differences which arose during the quenching treatment; namely, differences in stability and amount of retained austenite, differences

in quenching stress and differences in amount of ferrite and cementite particles.

In tempering above 572 degrees Fahr. (300 degrees Cent.) the change in density is a uniform increase showing the predominance of the third factor, resulting in a uniform growth of ferrite particles and martensite decomposition, until a temperature of 977 degrees Fahr. (525 degrees Cent.) is reached. Additional evidence of the influence of martensite decomposition is indicated by the nearly constant value of the slope of the curves representing the changes in density for the five quenching temperatures, which are given as follows:

Quenching Temperature Degrees Fahr.	Degrees Cent.	Slope of Density Curve for Centigrade Temperature
1600	(871)	7.7×10^{-5}
1700	(932)	7.9×10^{-5}
1800	(981)	7.9×10^{-5}
1900	(1038)	8.7×10^{-5}
2000	(1097)	9.1×10^{-5}

In tempering the bars quenched from 2000 degrees Fahr. (1097 degrees Cent.) between 977 degrees Fahr. (525 degrees Cent.) and 1121 degrees Fahr. (605 degrees Cent.) the density remains practically constant. This evidence points to the conclusion that the retained austenite decomposes during tempering within this temperature range and that the decomposition of martensite and austenite, factors three and four, proceed at rates which counterbalance each other as indicated by the small net change in density over this range of temperature. In tempering above 1121 degrees Fahr. (605 degrees Cent.) the slope of the curves which represent the change in density with tempering temperature, is approximately the same as the slope below 977 degrees Fahr. (525 degrees Cent.). This supports the view that martensite is a product of the decomposition of austenite during tempering and that the changes occurring in the magnet steel upon tempering are the same immediately above the range of temperature most favorable to the decomposition of austenite as immediately below that range.

Change in Rockwell C Hardness. The curves representing the change in hardness with tempering temperature are shown graphically in Fig. 2 for the specimens quenched from 2000 degrees Fahr. (1097 degrees Cent.). The changes in hardness during tempering of the quenched tungsten magnet steel give added confirmation to the conclusions already drawn from the density experiments. Practically full hardness was developed in the tungsten steel during quenching.

which is in agreement with the work of Mathews (3). The curves showing the change in hardness during tempering of the oil, water, and 5 per cent sodium hydroxide quenched bars fall close together over the whole range of tempering temperature. Little difference in hardness was observed in the three quenching mediums. The slope

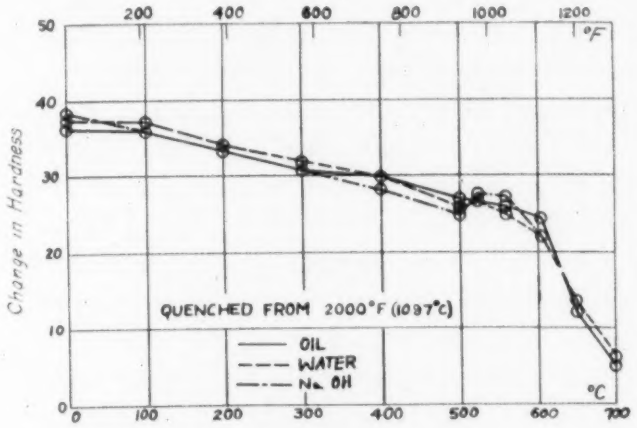


Fig. 2—Change in Rockwell C Hardness upon Tempering. Values Obtained in Annealed Condition Were Used as a Basis.

of the change in hardness curves is practically a constant between 212 degrees Fahr. (100 degrees Cent.) and 932 degrees Fahr. (500 degrees Cent.) as shown by the following table:

Quenching Temperature		Slope of Hardness Curve
Degrees Fahr.	Degrees Cent.	
1600	(871)	-3.3×10^{-2}
1700	(932)	-3.2×10^{-2}
1800	(983)	-3.0×10^{-2}
1900	(1038)	-2.7×10^{-2}
2000	(1097)	-2.7×10^{-2}

The constant slope of the change in hardness curves indicates that retained austenite is preserved during tempering until a temperature of 977 degrees Fahr. (525 degrees Cent.) is reached. The change in hardness curves for the lower quenching temperatures fall off more quickly with tempering temperature than those for the higher quenching temperatures upon increasing the temperature above 932 degrees Fahr. (500 degrees Cent.). In the bars quenched from 1900 degrees Fahr. (1038 degrees Cent.), and 2000 degrees Fahr. (1097 degrees Cent.) the phenomenon of secondary hardness, described by Bain (12), was noticeable. This points to a greater retention of austenite in the specimens quenched from the higher temperatures. The fact that retained austenite decomposes most readily during tempering

above 977 degrees Fahr. (525 degrees Cent.) is indicated by the secondary hardness effect, which substantiates the conclusions obtained from the changes in density, page 740.

Change in Magnetic Properties. Since the range of temperature most favorable to austenite decomposition during tempering has been located, it now remains to observe the change in magnetic properties which occur upon tempering within this range. The data for the curves representing the changes in magnetic properties were calculated from the values of maximum induction Bm, residual induction Br, and coercive force Hc, using as a basis the values of these properties obtained after an anneal of four hours at 1750 degrees Fahr. (955 degrees Cent.).

In Fig. 3 the curve representing the change in maximum induction Bm with tempering temperature is given for the specimens quenched from 2000 degrees Fahr. (1097 degrees Cent.). The maximum induction Bm, increases with tempering until a temperature of 572 degrees Fahr. (300 degrees Cent.) is reached. The value of Bm, remains approximately constant between 572 degrees Fahr. and 932 degrees Fahr. (500 degrees Cent.) for all quenching temperatures, 1600 degrees Fahr. (871 degrees Cent.) to 2000 degrees Fahr. (1097 degrees Cent.) inclusive. In tempering above 932 degrees Fahr. (500 degrees Cent.) Bm is observed to fall off, beginning as follows, for the different quenching temperatures:

Quenching Temperature		Bm Begins to Decrease Upon Tempering at	
Degrees Fahr.	Degrees Cent.	Degrees Cent.	Degrees Fahr.
1600	(871)	500	(932)
1700	(933)	500	(932)
1800	(983)	525	(977)
1900	(1038)	560	(1040)
2000	(1097)	605	(1121)

It is seen from the table that the maximum value of B max. is maintained over a longer range of tempering temperatures in the specimens quenched from the higher temperatures. This is analogous to the retention of hardness at higher temperatures in the specimens quenched from 2000 degrees Fahr. (1097 degrees Cent.), see page 741. The value of Bm is observed to fall off with the decomposition of retained austenite during tempering. Noticeable differences in maximum induction caused by quenching in different mediums, oil, water and 5 per cent sodium hydroxide, become less as the tempering temperature increases. Oil quenching was found to produce the lower value of Bm for each quenching temperature.

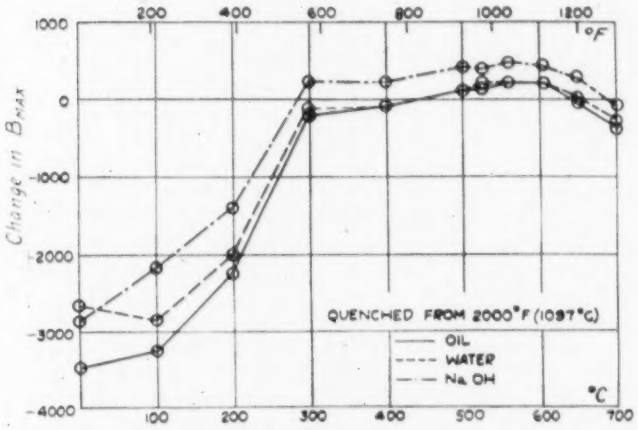


Fig. 3—Change in Maximum Induction Upon Tempering. Values Obtained in Annealed Condition Were Used as a Basis.

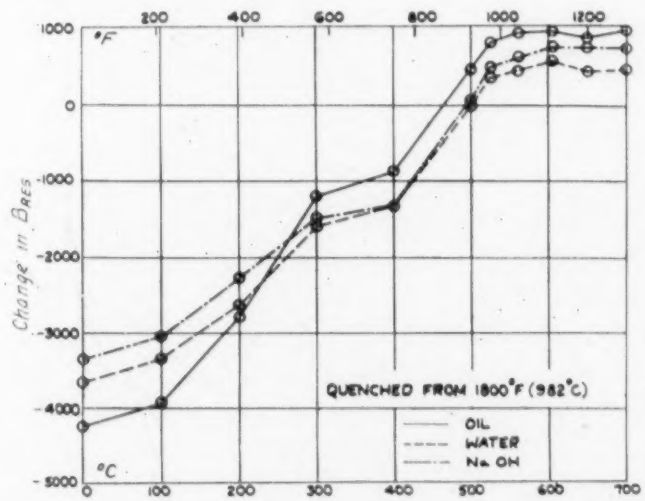


Fig. 4—Change in Residual Induction Upon Tempering. Values Obtained in Annealed Condition Were Used as a Basis.

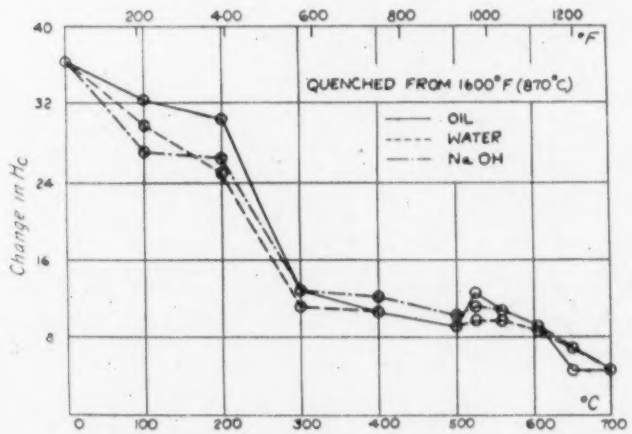


Fig. 5—Change in Coercive Force Upon Tempering. Values Obtained in Annealed Condition Were Used as a Basis.

In Fig. 4 the changes in residual induction are represented graphically for the specimens quenched from 1800 degrees Fahr. (983 degrees Cent.). From the curves it would appear that a very definite maximum in Br occurs upon tempering at 977 degrees Fahr. (525 degrees Cent.); or, in other words, austenite decomposition is accompanied by an increase in the value of the residual induction. Oil-quenched specimens in every instance gave the lower value of Br.

The change in coercive force, Hc, upon tempering is plotted in Fig. 5 for the specimens quenched from 1600 degrees Fahr. (871 degrees Cent.). The value of Hc decreases with tempering up to a temperature of 572 degrees Fahr. (300 degrees Cent.). The coercive force remains approximately constant from this point until the temperature reaches 932 degrees Fahr. (500 degrees Cent.). An increase in the value of Hc occurs for all quenching temperatures upon tempering at 977 degrees Fahr. (525 degrees Cent.). This change in the coercive force has the same direction as the change which is found to occur in the quenching treatment and indicates the formation of martensite as a result of austenite decomposition during tempering.

The change in the product Br × Hc, occurring upon tempering the quenched 5 per cent tungsten steel at successively higher temperatures, is shown in Fig. 6 for the bars quenched from 1800 degrees Fahr. (983 degrees Cent.). Upon tempering, the product Br × Hc shows an increase beginning at 977 degrees Fahr. (525 degrees Cent.) for all quenching temperatures.

The differences between the Br × Hc values for oil and water quenches and for oil and five per cent sodium hydroxide quenches are plotted in Fig. 7 with the quenching temperatures. It is clearly shown that a relationship exists, at least up to a quenching temperature of 1900 degrees Fahr. (1038 degrees Cent.), even though this may not be expressed mathematically.

Inspection of Fig. 7 shows that a very large difference in the Br × Hc values for oil and water quenches was obtained between the quenching temperatures, 1700 degrees Fahr. (933 degrees Cent.) and 1800 degrees Fahr. (983 degrees Cent.). Immediately above and immediately below, however, the difference in Br × Hc values for oil and water quenches tends to be a constant. The difference in Br × Hc value for the oil and water quenches shows a step decrease with quenching temperature.

The first change in the quotient Br/Hc during tempering is an increase, which becomes most marked in tempering between 392 degrees Fahr. (200 degrees Cent.) and 572 degrees Fahr. (300 degrees Cent.). Upon tempering at 977 degrees Fahr. (525 degrees Cent.) a marked decrease in Br/Hc occurs, as shown in Fig. 8 for the specimens quenched from 1600 degrees Fahr. (871 degrees Cent.). This

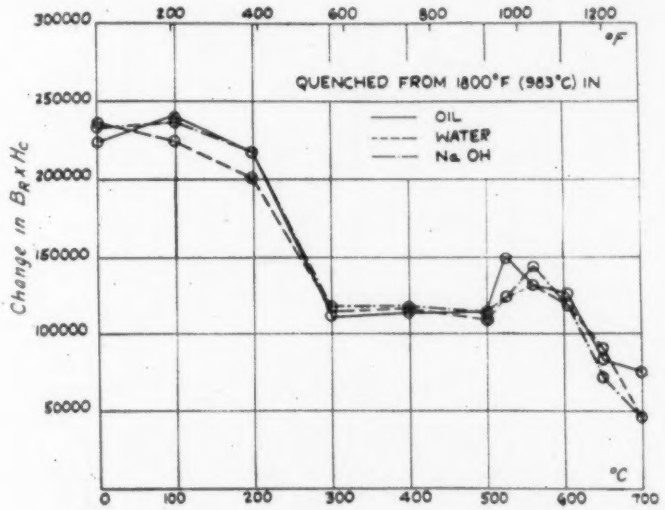


Fig. 6—Change in Product $B_r \times H_c$ Upon Tempering. Values Obtained in Annealed Condition Were Used as a Basis.

effect is most pronounced in the bars quenched from the lower temperatures. The value of Br/Hc for the oil quench is less than the value for the water or 5 per cent sodium hydroxide quenches up through a tempering temperature of 572 degrees Fahr. (300 degrees Cent.). Austenite decomposition during tempering is accompanied by a decrease in Br/Hc. This change has the same direction as the change in Br/Hc which occurred upon quenching.

In the quenched condition the difference in the value of Br/Hc between oil and water, and oil and 5 per cent sodium hydroxide quenched specimens shows a definite tendency to become a constant, the average value of which is 29 and 32, respectively. This relationship is brought out in the following table:

Quenching Temperature	1600° Fahr. (871° Cent.)	1700° Fahr. (933° Cent.)	1800° Fahr. (983° Cent.)	1900° Fahr. (1038° Cent.)	2000° Fahr. (1097° Cent.)	Average Value
Value of Br/Hc for oil	169	179	158	167	173	169
Value of Br/Hc for water	200	209	177	199	208	197
Difference	31	30	19	32	35	29
Value of Br/Hc for oil	169	179	158	167	173	171
Value of Br/Hc for 5% NaOH	199	198	191	214	203	201
Difference	30	19	33	47	30	32

It is believed that the water and NaOH quenches while differing from each other are sufficiently rapid in comparison with oil quenching that they lie on the same side of a critical rate of cooling which causes the nearly constant difference in Br/Hc values of oil and water, and oil and NaOH quenched specimens. Not only does

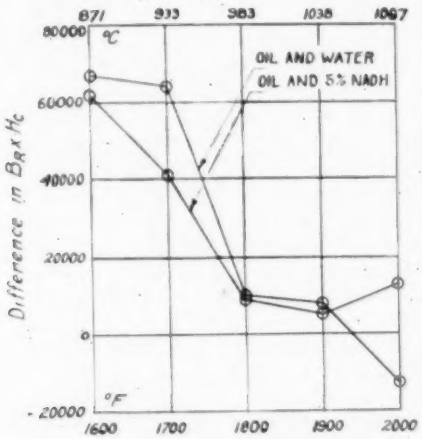


Fig. 7—Shows Difference in Product Br×Hc for Oil and Water, and Oil and 5 Per Cent NaOH Quenches for Different Quenching Temperatures.

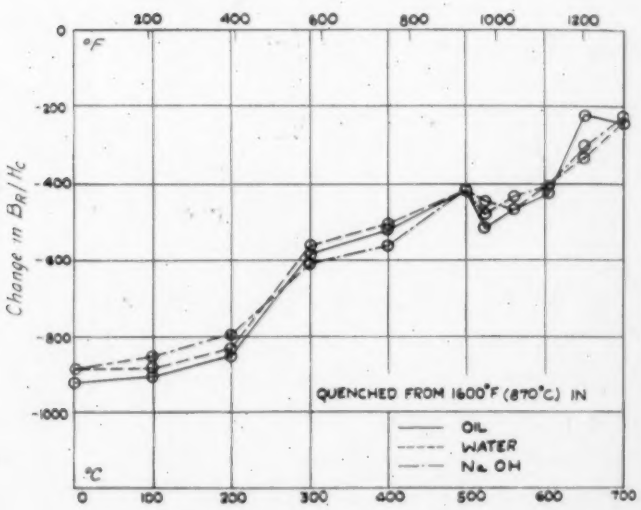


Fig. 8—Change in Quotient Br/Hc Upon Tempering. Values Obtained in Annealed Condition Were Used as a Basis.

the difference in the values of the quotient Br/Hc for oil and water, and oil and sodium hydroxide tend to become a constant; but also, the value of the quotient Br/Hc itself shows a definite tendency to become a constant independent of the quenching temperature.

If the maximum point on the permeability curve, which occurs between a magnetizing force of 15 and 70 Gilberts per centimeter, is plotted against the tempering temperature, the curves in Fig. 9 for oil-quenched bars are obtained. The permeability B/H was calculated from the normal induction data. From this figure it will be

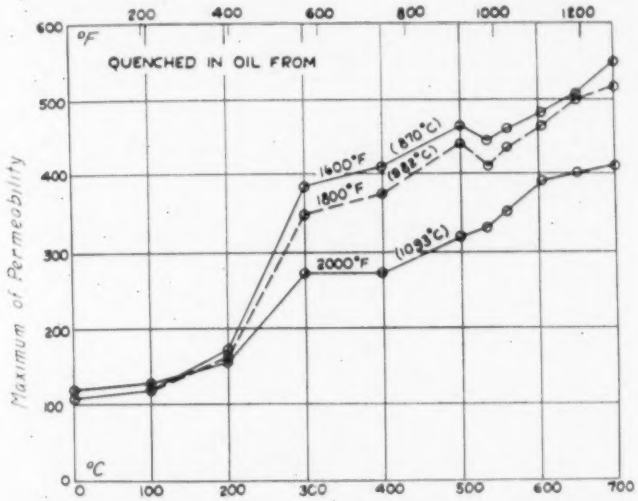


Fig. 9—Showing Maximum of Permeability with Tempering Temperature.

observed that a break in the maximum of permeability occurs upon tempering above 977 degrees Fahr. (525 degrees Cent.). This break is most pronounced, first, when the quenching is carried out at 1600 degrees Fahr. (871 degrees Cent.); and, second, when the quenching is carried out in oil.

Results of Liquid Air Experiments

The effect of the decomposition of retained austenite during immersion in liquid air upon the magnetic and other properties was studied by determining the change in density, the change in Rockwell C hardness and the changes in the magnetic properties with time of immersion in liquid air. The changes in properties during immersion in liquid air were calculated from the values observed, using as a basis the values obtained in the quenched condition for the graphical representation of the changes.

The changes in density upon immersion in liquid air are shown graphically in Figs. 10 and 11. The differences in behavior of the water and 5 per cent sodium hydroxide quenched specimens, which existed in the quenched condition, see page 739, were found to hold

throughout the treatments in liquid air. This indicates that the differences in behavior observed were dependent upon variables controlled by the quenching treatment.

It was shown previously that the greater change in density during quenching took place in the specimens quenched from 2000

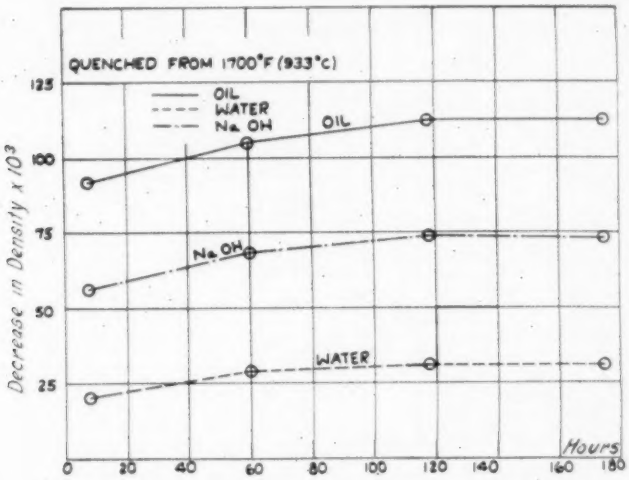


Fig. 10—Change in Density During Liquid Air Immersion. Values Obtained in Quenched Condition Were Used as a Basis.

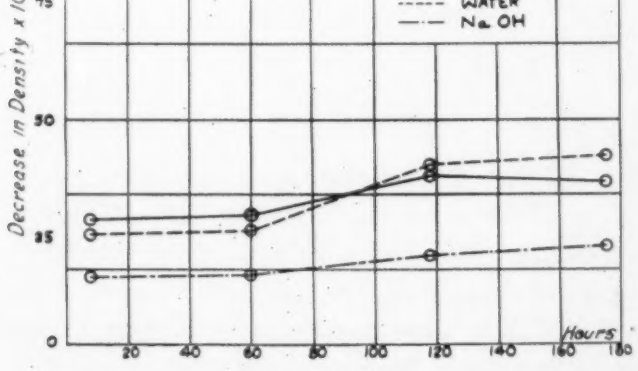


Fig. 11—Change in Density During Liquid Air Immersion. Values Obtained in Quenched Condition Were Used as a Basis.

degrees Fahr. (1097 degrees Cent.). On the other hand, when the quenched tungsten steel bars were given an immersion treatment in liquid air, the greater change in density took place in the bars quenched from 1600 degrees Fahr. (871 degrees Cent.). This dif-

ference in behavior of the bars quenched at the lower and higher quenching temperatures is indicative of the relative stability of austenite retained under these conditions. It is, therefore, indicated that quenching from the higher temperature results in a greater degree of stability for austenite which is subjected to immersion in liquid air.

The change in density in no instance was complete after eight hours immersion in liquid air, although the greater proportion of the total change occurred during the first eight hour immersion, as shown in the following table.

Quenching Temperature Degrees Fahr.	Degrees Cent.	Quenching Medium	Per cent of total change in density for 175 hour total immersion which occurs in first eight hour immersion. Per Cent
1700	(933)	Oil	82
		Water	65
		5 per cent NaOH	77
2000	(1097)	Oil	71
		Water	75
		5 per cent NaOH	70

If the density change may be an indication of the progress of austenite decomposition during immersion in liquid air, then this observation is not in agreement with the work of Bain and Waring (5) in which they conclude that "the uniform low temperature is apparently sufficient to destroy the austenite and further liquid air treatment accomplishes little or nothing."[1] It is shown that immersion in liquid air is not sufficient to decompose completely the austenite retained in the 5.70 per cent tungsten magnet steel. This fact is especially true at the higher quenching temperatures, shown by comparing the results obtained on a 1700 degrees Fahr. (933 degrees Cent.) quench and those where the 2000 degrees Fahr. (1097 degrees Cent.) quench was used.

Change in Rockwell C Hardness. Rockwell C hardness values are given in Tables II and IV for the quenched specimens and the specimens immersed in liquid air for a total period of 175 hours, respectively. An inspection of the tables shows that immersion in liquid air results in a decrease in hardness in nine instances out of fourteen. This observation is not in agreement with the values given by Mathews (3) for a 5 per cent tungsten steel. Two possible explanations are offered for this deviation: first, the specimens used in making the hardness measurements after immersion in liquid air were cut from different bars; and, second, the hardness values were

[1]Their initial period of immersion was between 12 and 16 hours.

Table III
Tabulated Experimental Findings

Immersed in Liquid Air for 8 Hours
H Max. = 200 Gilberts/cm.

Quenching Temperature	Quenching Medium	Gauss Bn	Gauss Br	Gil./cm. Hc	Br × Hc	Br/Hc	Density
1600° F (871° C)	Oil	.....	.....	...	.....	...	8.0667
	Water	.....	.....	...	.....	...	8.0610
	NaOH	.....	.....	...	.....	...	8.0602
1700° F (933° C)	Oil	14590	9010	48.2	434,000	187	8.0682
	Water	14920	8670	41.7	362,000	208	8.0660
	NaOH	14580	9410	44.5	419,000	211	8.0625
1800° F (983° C)	Oil	13300	8080	46.0	371,000	175	8.0684
	Water	13720	8900	44.6	397,000	200	8.0655
	NaOH	13940	8830	43.1	381,000	205	8.0624
1900° F (1038° C)	Oil	14080	8710	46.3	403,000	188	8.0627
	Water	14450	9520	43.9	418,000	217	8.0620
	NaOH	14710	9530	42.7	407,000	224	8.0595
2000° F (1097° C)	Oil	13720	8200	43.0	352,400	191	8.0621
	Water	14250	8550	41.6	356,000	206	8.0587
	NaOH	14430	8910	38.9	347,000	229	8.0578

Table IV
Tabulated Experimental Findings

Immersed in Liquid Air for 175 Hours
Hmax = 200 Gilberts/cm.

Quenching Temperature	Quenching Medium	Gauss Bm	Br	Gil/cm Hc	Br × Hc	Br/Hc	Density	Rockwell "C" Hardness
1600° F (871° C)	Oil	.....	....	...	.....	...	8.0643	63
	Water	.....	....	...	.....	...	8.0585	62
	NaOH	.....	....	...	.....	...	8.0576	62
1700° F (933° C)	Oil	14560	9200	48.5	446,000	190	8.0662	61
	Water	14840	8990	41.1	369,000	219	8.0649	61
	NaOH	14540	9350	47.1	449,000	203	8.0608	62
1800° F (983° C)	Oil	13350	8260	44.4	367,000	186	8.0670	61
	Water	13720	8980	44.1	396,000	204	8.0640	62
	NaOH	13950	8830	42.1	372,000	210	8.0608	60
1900° F (1038° C)	Oil	14080	8790	46.2	406,000	191	8.0617	63
	Water	14450	9500	44.8	426,000	212	8.0607	60
	NaOH	14680	9660	42.9	414,000	225	8.0585	64
2000° F (1097° C)	Oil	13740	8330	43.8	365,000	190	8.0605	62
	Water	14240	8650	40.3	349,000	215	8.0571	61
	NaOH	14440	8920	38.5	343,000	232	8.0568	..

taken about ten months after immersion in liquid air and aging at room temperature may have produced a difference in the steel bars which were quenched only, and those quenched, followed by a 175 hour immersion in liquid air.

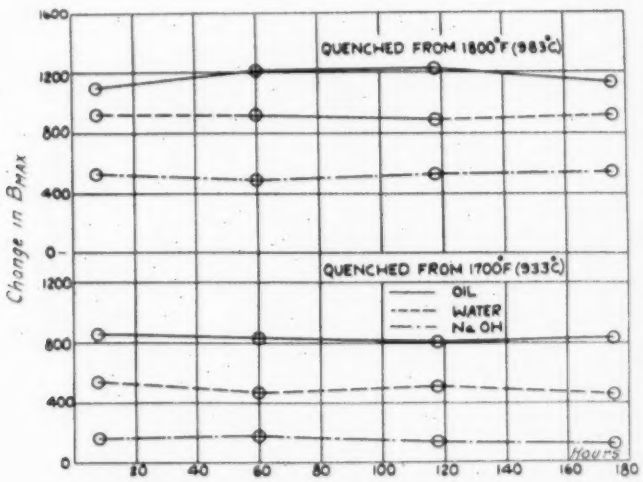


Fig. 12—Change in Maximum Induction During Liquid Air Immersion. Values Obtained in Quenched Condition Were Used as a Basis.

Change in Magnetic Properties. The changes in the magnetic properties caused by the decomposition of retained austenite in the quenched 5 per cent tungsten steel during immersion in liquid air have been observed at intervals over a total immersion of 175 hours. The properties studied consist of the changes in maximum induction (Bm), residual induction (Br), coercive force (Hc), the product (Br $\times$ Hc), the quotient (Br/Hc), and the permeability (B/H). The data for the curve representing the change in properties were calculated using the values obtained in the quenched condition as a basis.

The changes in Bm observed during immersion in liquid air are shown in Fig. 12 for the bars quenched from 1800 degrees Fahr. (983 degrees Cent.) and 1700 degrees Fahr. (933 degrees Cent.) for three quenching mediums: oil, water and 5 per cent sodium hydroxide. Immersion in liquid air results in an increase in the maximum induction for all quenching temperatures and for all the quenching mediums. This is in agreement with the work of Mathews, whose results are cited above. Prolonged immersion resulted in little further increase in the value of the maximum induction. On the other hand

it was shown, page 742, that the value of Bm decreased with the decomposition of austenite during tempering.

In the bars quenched at 1700 degrees Fahr. (933 degrees Cent.) to 1900 degrees Fahr. (1038 degrees Cent.), inclusive, the change in Bm upon immersion in liquid air is greater in the oil-quenched bars

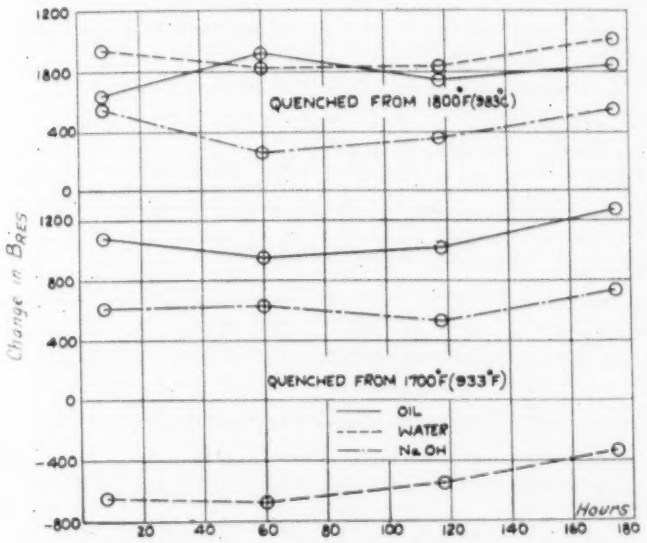


Fig. 13—Change in Residual Induction During Immersion in Liquid Air. Values for Quenched Condition Were Used as a Basis.

and less in the bars quenched in 5 per cent sodium hydroxide; while at 2000 degrees Fahr. (1097 degrees Cent.) the bars quenched in oil and 5 per cent sodium hydroxide show about the same change in Bm upon immersion in liquid air. The maximum change was produced by quenching in oil from 1800 degrees Fahr. (983 degrees Cent.), while the smallest change occurred in the bars quenched from 1700 degrees Fahr. (933 degrees Cent.) in sodium hydroxide.

The graphical representations of the change in the residual induction Br upon immersion in liquid air are given in Fig. 13 for the specimens quenched in the lower temperatures. In all instances, except the specimen quenched in water from 1700 degrees Fahr. (933 degrees Cent.), an increase in the residual induction, Br, was observed. This is in agreement with the results of Mathews previously cited and the results of Bain and Waring (5). Prolonged immersion resulted in slight further increase in the value of Br in most instances. The oil-quenched bars show the greater change in

Br upon immersion in liquid air except those quenched from 1800 degrees Fahr. (983 degrees Cent.). Similar to the change in density during immersion in liquid air, the greater change in Br occurs in the bars quenched from the lower temperatures, while the smaller

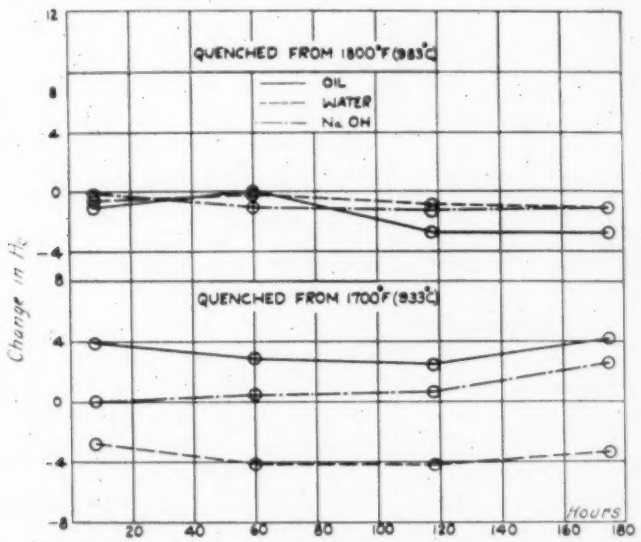


Fig. 14—Change in Coercive Force During Immersion in Liquid Air. Values for Quenched Condition Were Used as a Basis.

change in Br occurred in the specimens quenched from 2000 degrees Fahr. (1097 degrees Cent.).

In Fig. 14 are shown the changes in coercive force, produced by immersion in liquid air. In eight instances out of twelve the coercive force decreases upon immersion of the quenched bars in liquid air. This is in agreement with the results of Mathews. On the other hand, Bain and Waring (5) obtained an increase in coercive force upon immersion for a period of twelve hours; however, their results are for a low manganese steel and may not be comparable with the results just described. The decomposition of austenite during tempering was shown, page 744, to give an increase in coercive force. This striking difference in behavior is indicative of a difference in the mode of decomposition depending on whether the austenite decomposition is effected by tempering or by liquid immersion.

Immersion in liquid air gave an increase in the product Br × Hc with an exception of one instance in which the specimen was quenched in 5 per cent sodium hydroxide from 2000 degrees Fahr. (1097 de-

gress Cent.). The greater change in the product Br × Hc upon immersion in liquid air occurred with the bars quenched from the lower temperature, 1700 degrees Fahr. (933 degrees Cent.), which is similar to the change in density and to the change in residual induction.

The value of Br/Hc showed an increase after immersion in liquid air in all instances, except the specimen quenched in water from 2000 degrees Fahr. (1097 degrees Cent.). This observation is in agreement with the changes given by Mathews (3). When the period of

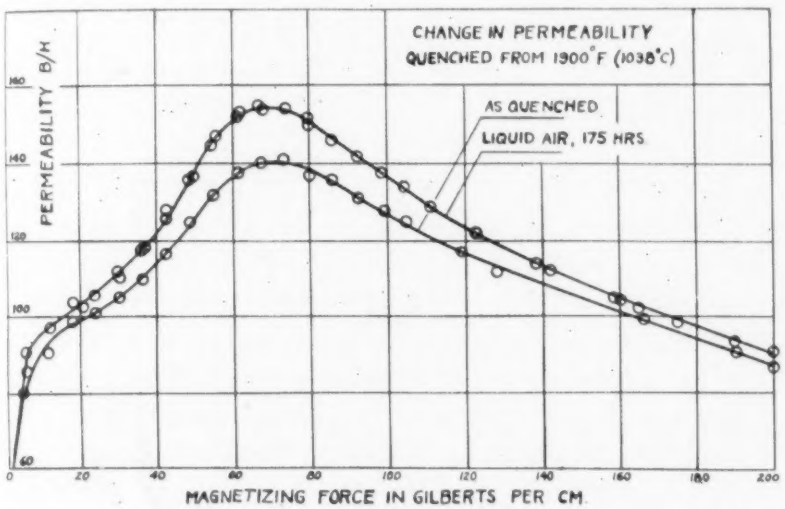


Fig. 15—Change in Permeability During Immersion in Liquid Air.

immersion exceeded sixty hours, the oil-quenched bars showed the greater change in the quotient Br/Hc except in the 2000 degree Fahr. (1097 degree Cent.) quench. The same type of relationship exists throughout the liquid air treatments, as was observed in the quenched condition between the quotient Br/Hc and quenching medium. The oil-quenched specimens show lower values of Br/Hc than either the water-quenched bars or those quenched in 5 per cent sodium hydroxide. When the quenching is carried out at 1800 degrees Fahr. (983 degrees Cent.) and above, the value of Br/Hc in the quenched condition and after liquid air treatment is lower for the oil and higher for the specimens quenched in 5 per cent sodium hydroxide.

In Fig. 15 the change in permeability B/H is given for a total of 175 hours immersion in liquid air and shows that the permeability is increased by liquid air immersion. This is directly opposite to the effect produced upon decomposing austenite by tempering at 977 degrees Fahr. (525 degrees Cent.), where a noticeable decrease in the

maximum permeability was observed. This reversal in the effect is not limited to permeability, as is evident in the following table, giving the observed changes in properties of the quenched 5 per cent tungsten steel resulting from austenite decomposition during tempering and during immersion in liquid air.

Property Observed	Change Within Tempering Temperature Range 932°F (525°C) to 1121°F (605°C)	Change Upon Immersion in Liquid Air
Density	decrease	decrease
Hardness	increase	decrease
Maximum induction Bm	decrease	increase
Residual Induction Br	increase	increase
Coercive force Hc	increase	decrease
Product Br x Hc	increase	increase
Quotient Br/Hc	decrease	increase
Maximum permeability B/H	decrease	increase
Permeability B/H	decrease	increase

Austenite decomposition during tempering and upon immersion in liquid air gave opposite changes in the values of hardness, maximum induction, coercive force quotient Br/Hc and the permeability. These differences in the effect of austenite decomposition on the properties, depending upon whether the decomposition is accomplished by liquid air immersion or by tempering is believed to be the result of the mode of decomposition on the effectiveness of the five factors outlined previously. When austenite decomposes during tempering the stresses set up within the specimens by quenching are relieved. The decomposition proceeds in tempering more slowly, giving time for the precipitation of cementite and subsequent coalescence of the particles. During tempering the growth of ferrite particles also takes place as the temperature is increased. The decomposition of martensite and troostite accompanies the decomposition of austenite during tempering within the range, 977 degrees Fahr. (525 degrees Cent.) to 1121 degrees Fahr. (605 degrees Cent.). On the other hand, when the retained austenite decomposes during immersion in liquid air the quenching stresses are still present and the coalescence of cementite and growth of ferrite particles is impeded by the low temperature. The rate of decomposition of austenite is rapid during immersion in liquid air as indicated by the greater part of the total change taking place within the first eight hour period of immersion.

Summary

From the present investigation of the effect of the decomposition of austenite retained in a 5 per cent tungsten steel on the change in

magnetic properties certain definite relationships have been established. These relationships, however, are not of such a character that they permit mathematical formulations between the amount of austenite present after any given heat treatment and the magnetic properties.

More austenite was retained in the specimens quenched from the higher temperatures as indicated, first, by the greater density change upon quenching; and, second, by the secondary hardness effect during tempering, in the bars quenched from 2000 degrees Fahr. (1097 degrees Cent.).

The bars quenched in oil showed a greater density than did the bars quenched in water or 5 per cent sodium hydroxide. Bars quenched in water from 1800 degrees Fahr. (983 degrees Cent.) or above showed a greater density than did the bars quenched in 5 per cent sodium hydroxide. On the other hand, bars quenched in 5 per cent sodium hydroxide from below 1800 degrees Fahr. gave a greater density than did the bars quenched in water.

Austenite retained in the quenched tungsten steel was found to decompose most readily upon tempering within the temperature range 977 degrees Fahr. (523 degrees Cent.) to 1121 degrees Fahr. (605 degrees Cent.). The changes in magnetic properties which occurred upon tempering within this temperature range are the result of the decomposition of austenite in the quenched steel.

An apparent relation exists between the product Br × Hc and the quenching temperature but only in the quenched condition.

A definite relationship between the quotient Br/Hc and the quenching temperature was found to exist only in the quenched condition. The Br/Hc value for oil-quenched bars is consistently lower than the value for the bars quenched in water or 5 per cent sodium hydroxide by an amount which tends to become a constant. The value Br/Hc, also, shows a remarkable tendency to become a constant. Oil quenching produces more retained austenite than either water or 5 per cent sodium hydroxide quenches. These two findings are in agreement with the results of previous investigators.

A 5.70 per cent tungsten steel, containing 0.60 per cent carbon, quenched in oil gave a lower value of maximum induction (Bm), residual (Br), product (Br × Hc) and quotient (Br/Hc) than when quenched in water or 5 per cent sodium hydroxide. Oil-quenched specimens gave higher value of coercive force (Hc) and a greater

density for all quenching temperatures 1600 degrees Fahr. (871 degrees Cent.) to 2000 degrees Fahr. (1097 degrees Cent.), inclusive.

The greater change in density of the quenched tungsten steel bars upon immersion in liquid air was found to occur, first, when the quenching took place from the lower temperatures; and, second, when the quenching was carried out in oil.

The decomposition of the austenite by immersion in liquid air, while taking place largely within the first eight hours, was gradually increased with prolonged immersion.

The decomposition of austenite by immersion in liquid air was influenced by the quenching temperatures. Austenite retained in the bars quenched from the higher temperatures showed a greater degree of stability upon immersion in liquid air than did the austenite retained by quenching from the lower temperatures.

The effects of the decomposition of austenite in the quenched steel on the magnetic properties are summarized in the following outline, together with the changes in density and Rockwell C hardness.

Property	Decomposition of Austenite During Drawing	Decomposition of Austenite Upon Immersion in Liquid Air
Density	decrease	decrease
Hardness	increase	decrease
Maximum inductance Bm	decrease	increase
Residual inductance Br	increase	increase
Coercive force Hc	increase	decrease
Product Br x Hc	increase	increase
Quotient Br/Hc	decrease	increase
Maximum of permeability B/H	decrease	increase
Permeability B/H	.....	increase

The observed differences in effect of austenite decomposition depending on whether the decomposition is effected by liquid air immersion or by tempering are believed to be the result of mode of decomposition on the effectiveness of certain factors in liquid air treatment which come into normal effectiveness during decomposition upon tempering. Decomposition during tempering follows the austenite-martensite-troostite-sorbite type of change while in liquid air the decomposition simulated the austenite-troostite change without the accompanying growth of cementite and ferrite particles.

Bibliography

1. Dowdell, R. L., and Harder, O. E., "The Decomposition of the Austenitic Structure in Steel", Transactions, American Society for Steel Treating, Vol. 11, 1927, p. 17-41.

2. Nusbaum, C., "Certain Aspects of Magnetic Analysis", *Proceedings*, American Society for Testing Materials, Vol. 19, part II, 1919, p. 96-116.
3. Mathews, J. A., "Retained Austenite; A Contribution to the Metallurgy of Magnetism", TRANSACTIONS, American Society for Steel Treating, Vol. 8, 1925, p. 565-583.
4. Dowdell, R. L., and Harder, O. E., "The Decomposition of the Austenitic Structure in Steels", part II, "The Decomposition of Austenite in Liquid Oxygen", TRANSACTIONS, American Society for Steel Treating, Vol, 11, 1927, p. 391-398.
5. Bain, E. C., and Waring, W. S. N., "Austenite Decomposition and Length Changes in Steel", TRANSACTIONS, American Society for Steel Treating, Vol. 15, 1929, p. 69-95.
6. Honda, K., and Iwase, K., "On the Transformation of Retained Austenite into Martensite by Stress", TRANSACTIONS, American Society for Steel Treating, Vol. 11, 1927, p. 399-412.
7. Honda, K., and Osawa, A., "On the Distribution of Austenite in Specimens of quenched Carbon Steels", *Science Reports*, Tohoku Imperial University, Sendai, Japan, Vol. 18, 1929, p. 47.
8. Tamman, G., and Scheil, E., "Die Umwandlungen des Austenit und Martensit in Gehärten Stählen", *Zeitschrift für Anorganische und Allgemeine Chemie*, Vol. 157, 1927, p. 1-21.
9. Schroeter, K., "Uber die Umwandlung des Austenit in Martensit durch flussige Luft", *Zeitschrift fur Anarganische und Allgemeine Chemie*, Leipzig, Vol. 169, 1928, p. 157-160.
10. Maurer, E., and Schroeter, K., "Die Bestimmung des Austenitgehaltes durch Messung des magnetischen Sättigungswertes und die Vorgänge beim Anlassen gehärteter Stähle", *Stahl und Eisen*, Vol. 49, 1929, p. 929-941.
11. Nurbury, A. L., "Volumes Occupied by the Solute Atoms in Certain Metallic Solid Solutions and their Consequent Hardening Effects", *Transactions*, Faraday Society, Vol. 19, 1924, p. 586.
12. Bain, E. C., "Secondary Hardness in Austenitized High Carbon-Chromium Steels", TRANSACTIONS, American Society for Steel Treating, Vol. 5, 1923, p. 89-101.

A STUDY OF THE IRON-CHROMIUM-CARBON CONSTITUTIONAL DIAGRAM

By V. N. Krivobok and M. A. Grossmann

Abstract

The present paper examines the changes in the microstructure of iron-chromium-carbon alloys, caused by variations in the chromium content with various carbon contents. Many photomicrographs of the various alloys studied are reproduced and tabulations presented of the microconstituents found at different temperatures. Diagrams are also included showing structures found in different alloys. Ternary diagrams up to 33 per cent chromium are reproduced.

Previous work has established the relationships that exist when the chromium content is varied from zero up to about 18 per cent. It had been shown, first, that in the absence of carbon an increase in chromium caused the region of delta iron to merge with the alpha region, eliminating gamma iron when the chromium content was above 14 per cent; and second, that in the presence of carbon the range of temperature and carbon content in which austenite was associated with delta iron merged with the range in which austenite was associated with alpha iron.

In the present paper, the alloys are examined further up to a chromium content of 35 per cent and a carbon content of 0.60 per cent. It is shown that the region of mixed structures [alpha (delta) iron plus austenite] is an extensive feature in the whole range from 20 to 35 per cent chromium, and also that the region of pure austenite is greatly reduced in extent.

It was found that the structures obtained at various temperatures in the different alloys can be related quite simply in a ternary constitutional diagram. A section of this diagram is constructed.

The addendum to this paper by Dr. V. N. Krivobok is a study of the iron-chromium-carbon alloys by means of thermal analysis. Each alloy previously studied metallographically was so analyzed. Typical curves of the results obtained are reproduced.

A paper presented before the Eleventh Annual Convention of the society in Cleveland, September 9 to 13, 1929. Of the authors, who are members of the society, Dr. V. N. Krivobok is associate professor of metallurgy at Carnegie Institute of Technology at Pittsburgh and Dr. M. A. Grossmann is metallurgical engineer, Republic Steel Corporation, Canton, Ohio. Manuscript received June 6, 1929.

Introduction

THE addition of chromium to iron and to iron-carbon alloys is well known to affect their structures profoundly. The new structures are in some cases present permanently while in other cases they develop only upon heat treatment. It is the purpose of this paper to offer data on a range of chromium-iron-carbon alloys which have hitherto been studied only inadequately, and to show that (in the range of alloys studied) comparatively simple relationships obtain among the structures which may be developed.

The alloys studied ranged from 20 to 35 per cent chromium and

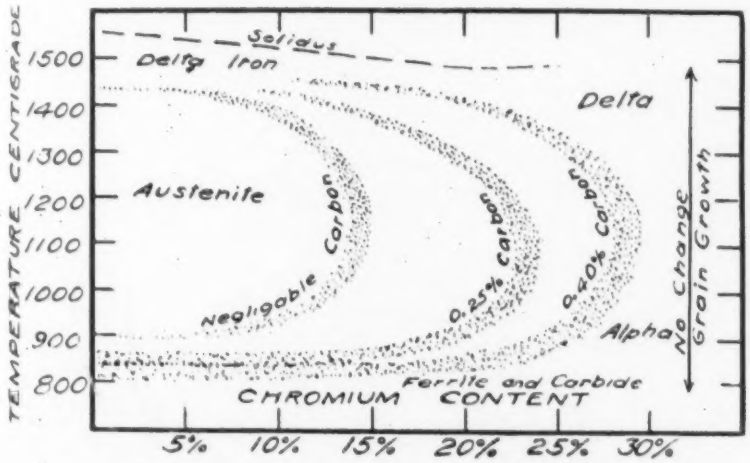


Fig. 1—Effect of Chromium on Gamma Transformation. (Bain).

from 0.10 to 0.60 per cent carbon, the balance is substantially iron. The object was to discover the structures developed by varying heat treatments, or (stated more precisely for this case) the structures existing when the alloys were heated at various elevated temperatures, these structures being preserved to room temperature by quenching.

Previous Work

Previous work suggested the lines along which this research might profitably be carried out. Bain[1] discovered the fact that the addition of about 13 per cent chromium to carbonless iron suppressed the gamma transformation, so that the alpha and delta regions were completely merged. Iron-chromium alloys with more than 13 per

[1]E. C. Bain, "The Nature of the Alloys of Iron and Chromium," TRANSACTIONS, American Society for Steel Treating, Vol. 9, 1926, p. 9.

cent chromium (in the absence of carbon) thus consist of alpha iron only, in all conditions of temperature and heat treatment. Bain's diagram is shown in Fig. 1. His data were obtained by quenching the alloys from the high temperatures and examining the preserved microstructures. Esser[2] examined a similar range of alloys with a differential thermocouple of his own design, and with a special dilatometer, and derived the diagram shown in Fig. 2. Maurer[3], using

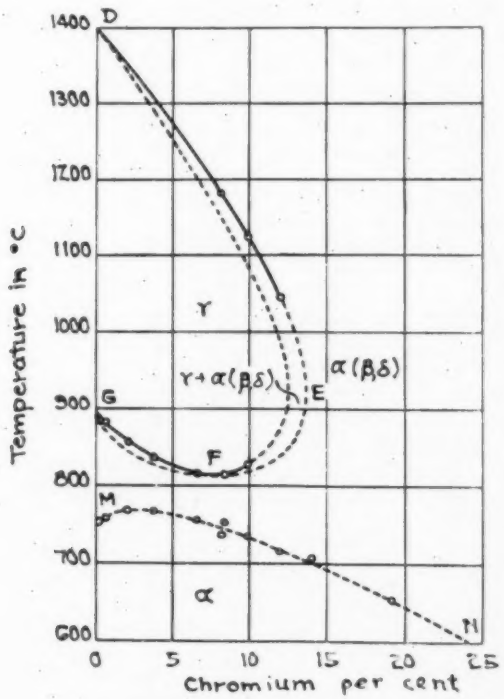


Fig. 2—Effect of Chromium on Gamma Transformation and on Magnetic Change. (Esser).

a Saladin differential thermocouple recorder, obtained the diagram of Fig. 3 for alloys containing very small amounts of carbon. Kinzel[4] observed length changes in fine wires and so located critical points for the diagram of Fig. 4. For the purposes of the present paper, it has been assumed that the average conditions are about as shown in Fig. 5.

The structures in the presence of carbon have been examined

[2]Esser and Oberhoffer, *Stahl und Eisen*, Vol. 47, 1927, p. 2026.

[3]Maurer and Nienhaus, *Stahl und Eisen*, Vol. 48, 1928, p. 996.

[4]A. B. Kinzel, "Critical Points in Chromium-Iron Alloys," *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 76, C28, (Iron and Steel Technology in 1928, p. 301).

much less completely. Carbon favors the formation of austenite, and therefore may be said to work counter to the effect of chromium in this respect. In the range beyond 13 per cent chromium, then, it becomes necessary to study the effect of carbon in inducing the presence of austenite despite the chromium.

It has been shown[5] that in the range just beyond 13 per cent chromium, mixtures of alpha iron and austenite are found in propor-

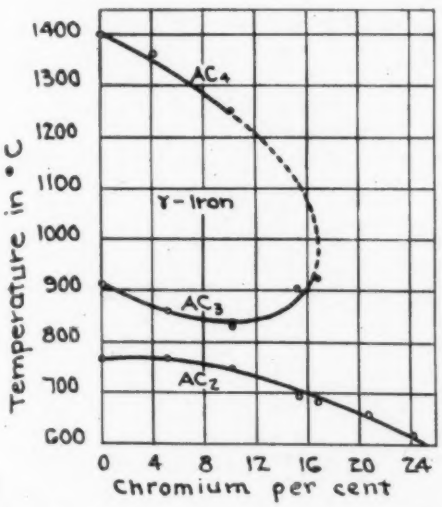


Fig. 3—Effect of Chromium on Gamma Transformation and on Magnetic Change. (Maurer).

tions that can be predicted. It was shown that not only does the delta region merge with the alpha region, but that further the range of composition and temperature in which austenite coexists with delta iron merges with the region in which austenite coexists with alpha iron. A plane section of the ternary diagram, at 18 per cent chromium, thus has the appearance shown[5] in Fig. 6. Westgren[6], Phragmen and Negresco independently arrived at the same conclusion, as shown in Fig. 7.

The action of chromium in altering the relationships in the iron-carbon diagram may be pictured as shown in Figs. 8 and 9. Fig. 8 indicates the relation of the iron-carbon diagram to the iron-chromium diagram in the ternary alloys. Sections of the ternary diagram at 5, 10, 14 and 18 per cent chromium would have the ap-

[5]M. A. Grossmann, "Nature of the Chromium-Iron-Carbon Diagram," *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 75, 1927, p. 214.

[6]Arne Westgren, Gosta Phragmen and Tr. Negresco, "On the Structure of the Iron-Chromium-Carbon System," *Journal*, Iron and Steel Institute, Vol. 117, 1928-1, p. 394.

pearance shown in Fig. 9. With increase in chromium content, the cross-hatched region above, where austenite is associated with delta iron, is seen to approach the cross-hatched region below, where austenite is in equilibrium with alpha iron, and finally to be merged with it.

Attention is directed to the fact that there is indicated a certain solubility of carbon in alpha iron as well as in delta iron.

We come now to the study of the alloys containing from 20 to 35 per cent chromium, with varying carbon content. After some deliberation, it was decided to adopt the following procedure to secure

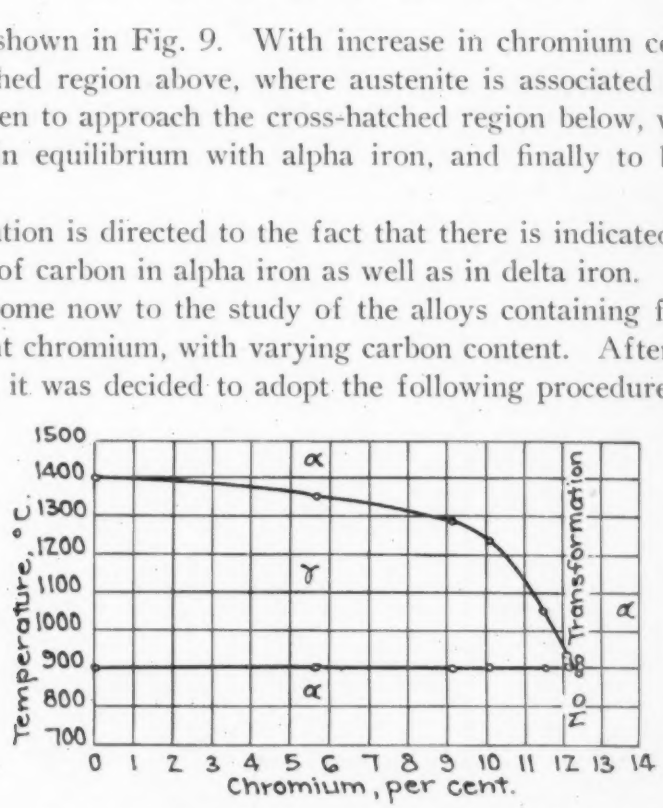


Fig. 4—Transformation Points in Pure Chromium-Iron Alloys. (Kinzel).

the structural constituents (and also of course, properties) as representative of the gradually increasing temperatures, because the alloys under study have been found to be readily retained in the metastable state (by drastic quenching):

> Heating the samples (after preliminary annealing) to a desired temperature, holding them there for a certain length of time and drastically quenching in a suitable quenching medium.

This procedure was carried out in a small globar type furnace with the corner compartment of heat resisting tube, practically of the size of the samples, closed at both ends. The use of this furnace was prompted by several important considerations: first, it permitted very accurate control of temperature with the temperature gradient practically negligible over the length of about 4 inches; secondly, it facilitated very quick quenching; and thirdly, what was quite important, it greatly reduced the decarburization of the sample due to the small

chamber of the furnace. The last difficulty was also taken care of by sectioning of the samples after final heat treatment and therefore examining the center of the treated sample.

The presentation of the evidence might be facilitated by the introduction of a short summary of metallographic terms and illustrations used in this study.

(a) *Carbides*—iron-chromium-carbon chemical compounds, without any attempt to either distinguish or classify them according to their formulae. Quite hard, as evidenced by the scratch test. Probably nonmagnetic. In annealed samples found in massive

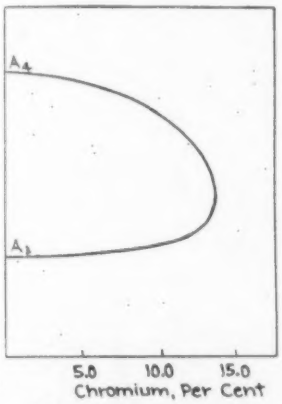


Fig. 5—Effect of Chromium on the Gamma Transformation on Heating.

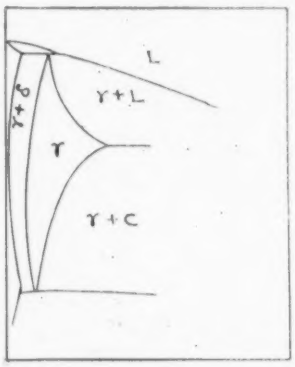


Fig. 6 — Section of Chromium - Iron - Carbon Diagram at 18 Per Cent Chromium. (Grossmann).

agglomerations. When etched with warm aqua regia these carbides are clearly outlined, without, however, etching of carbides proper. Murakami reagent darkens these carbides very readily in 10 to 30 seconds. (Photomicrographs Figs. 10 and 11.)

(b) *Alpha Iron*—magnetic chromium ferrite usually found in annealed alloys or in those that were quenched from below 1800 -1900 degrees Fahr. Always associated with carbides. Extremely difficult to polish and etch. Body-centered cubic atomic arrangement.

(c) *Delta Iron*—similar to alpha magnetic chromium ferrite, but found after quenching from high temperatures. Soft. Upon etching with aqua regia often displays a mottled appearance (Fig. 12) in contrast to structureless gamma iron, rendering the recognition of either one quite simple especially under high magnification. (Fig. 13). Is not etched by Murakami's reagent

When cold-worked the grains of delta iron always give the appearance of wavy lines, very much like the well known lines in cold-worked ferrite. (Figs. 14 and 15). When present together with gamma iron and carbides the latter are never found in delta, but are always imbedded in gamma. Magnetic. Body-centered cubic atomic arrangement with unit cube parameter of 2.86Å.*

(d) *Gamma Iron*—chromium iron with face-centered cubic arrangement of atoms and parameter of 3.59Å.* Nonmagnetic. After

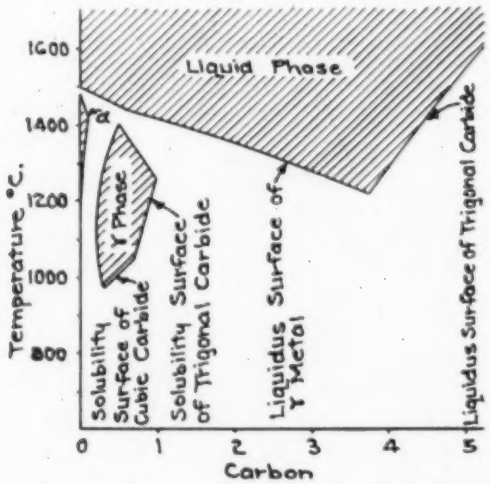


Fig. 7—Diagram Showing the Merging at the Regions in which Alpha Iron is Associated with Austenite. (After Westgren, Phragmen and Negresco.)

a scratch test appears to be harder than alpha or delta iron. Microscopically can be distinguished by white structureless appearance, (Fig. 13), often with distinct twinnings, (Fig. 29). If the twinnings are not seen on a polished and etched sample, slight cold working will bring them out quite clearly, (Fig. 16). In certain cases, however, twins are replaced by strain lines which are almost perfectly straight. In many cases gamma iron (or chromium austenite), (Fig. 15) grains are associated with carbide particles.

(e) *Liquefied metal*—that portion of the material which has become actually melted in the heat treatment. Sometimes it contains portions of typical eutectic appearance (Fig. 19), while at times,

*These X-ray studies were made by James Friauf, Ph.D., Bureau of Metallurgical Research, Carnegie Institute of Technology, Pittsburgh.

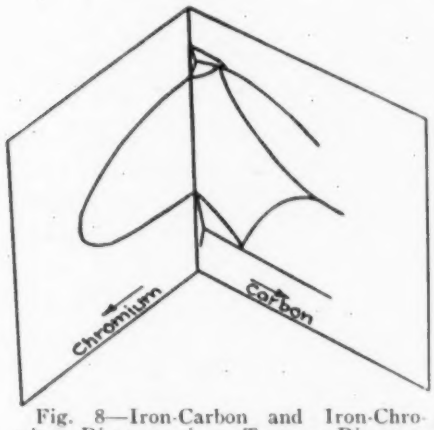


Fig. 8—Iron-Carbon and Iron-Chromium Diagrams in a Ternary Diagram.

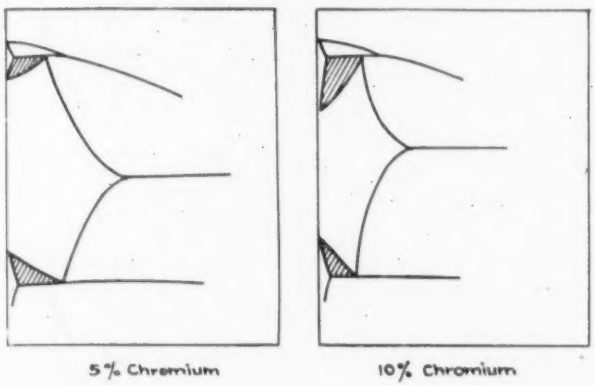


Fig. 9—Sections of Ternary Iron-Chromium-Carbon Diagram at Various Chromium Contents. (Grossmann.)

it presents more the mottled and indefinite structure of rapidly solidified mixtures of gamma and eutectic, (Fig. 17 and 18). Is found only in alloys heated to high temperature, probably exceeding 2350 degrees Fahr., (1290 degrees Cent.). First ap-

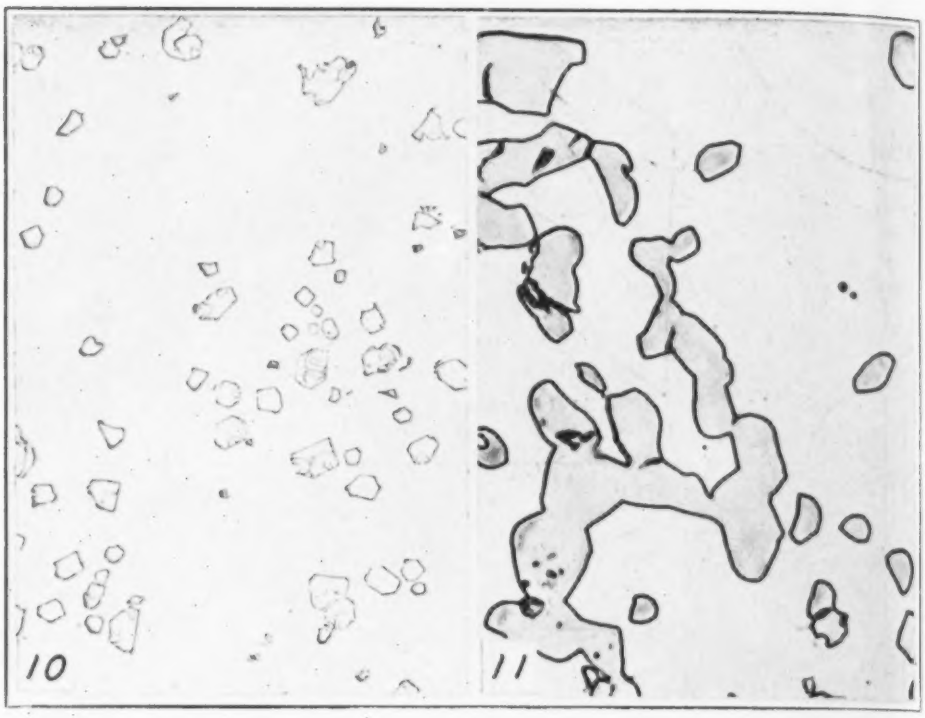


Fig. 10—Typical Carbides in Alpha or Delta Iron. Etched Aqua Regia. × 200.
Fig. 11—Typical Carbides in Alpha or Delta Iron. Etched Murakami's Reagent. × 2000.

pears at the grain boundaries. The structure of the eutectic is developed much better on slow cooling with a tendency on part of the constituents to separate out and agglomerate.

(f) *Chromium-troostite*—a constituent, which, as name implies, is in appearance very much like the troostite in carbon steels. It has been found in certain alloys only and represents, it is believed, one of the earliest stages in the separation of carbides in the form of very minute particles. Much harder than either delta iron or chromium austenite. Readily etched out by Murakami's reagent and somewhat less so by aqua regia. (Fig. 20).

Experimental Results

Description of Metallographic Data

The experimental results are recorded in Table I in such a way as to indicate the structural constituents of the alloys which are existant at different temperatures. Each table refers to the alloys of a narrow range of chromium content, and thus shows for this par-

Table I
Chromium Content 20 to 22 Per Cent
Summary of Constituents at Different Temperatures

Temperature	Carbon Content Per Cent 0.15	0.19	0.35	0.62
2500° F. 1370° C.	Large α (or δ) grains with numerous γ grains in Widmanstatten patterns. Magnetic. Fig. 33.	Large grains of α (or δ) containing some small γ grainlets in Widmanstatten patterns. Magnetic.	γ iron predominates. Some δ iron. Liquefied metal in isolated spots. No free carbides. Very slightly magnetic.	γ iron grains. Liquefied metal at grain boundaries and within grains. Nonmagnetic.
2400° F. 1315° C.	α (or δ) and γ iron grains. In certain places small γ grains assume Widmanstatten patterns. Magnetic. Fig. 31.	Large grains of α (or δ) and γ iron. Magnetic.	γ iron predominates. Some δ iron. No free carbides. Very slightly magnetic.	γ iron grains. Liquefied metal at grain boundaries. Nonmagnetic.
2300° F. 1260° C.	α (or δ) and γ iron in large grains. In some places δ grains contain very small γ platelets. Magnetic. Fig. 30.	Grains of α (or δ) and γ iron, the latter with numerous twins. The size of the grains is larger than at 2200° Fahr. Magnetic.	Largely γ iron with numerous twinnings. A few δ iron grains. Very slightly magnetic.	γ iron only. No free carbides.
2200° F. 1205° C.	Carbides disappear. The structure consists of α (or δ) and γ grains. The latter show twinning. Magnetic. Fig. 26.	Carbides disappear entirely. α (or δ) and γ iron grains, the latter with characteristic twinnings. Magnetic.	γ iron with numerous twinnings. Some δ iron. No carbides. Slightly magnetic.	Some free carbides in the well-developed austenitic grains. Nonmagnetic.
2100° F. 1150° C.	Large polyhedral grains of δ (or α) iron. Some γ iron, and free carbides much less in number. Magnetic. Fig. 25.	α (or δ) and γ iron grains. Very few free carbides. Magnetic.	γ iron with characteristic twinnings. Some δ iron grains. No carbides. Slightly magnetic.	Plentiful free carbides in the matrix of γ iron. Numerous twins. Nonmagnetic.
2000° F. 1095° C.	Polyhedral grains of α (or δ) iron still larger. Free carbides. Strongly magnetic. Fig. 21, 22, 23.	Grains of α (or δ) iron and γ iron. The former predominates. The latter with typical twinnings. Free carbides.** Strongly magnetic.	Free carbides. δ (or α) iron and γ grains. Slightly magnetic.	Plentiful free carbides in the matrix of γ iron. Numerous twins. Nonmagnetic.
1900° F. 1040° C.	Large polyhedral grains of α (or δ) iron and free carbides. Strongly magnetic.	Fairly large grains of α (or δ) iron. Some γ iron and free carbides. Strongly magnetic.	Plentiful free carbides. δ (or α) iron and γ grains. Slightly magnetic.	Plentiful free carbides in the matrix of α (or δ) or γ iron. Very slightly magnetic.
1800° F. 985° C.	Polyhedral grains of α (or δ) iron and numerous free carbides. Strongly magnetic.	Small grains of α (or δ) iron and very numerous carbides.* Strongly magnetic.	δ (or α) iron predominates. Numerous carbides and a few isolated γ iron grains. Magnetic.	Very large number of very fine carbides. α (or δ) iron matrix. Magnetic.
Alloy No.	2235	2210	468	469

*When heating time is increased to about 20 or 25 hours, the alloy shows the appearance of a few γ grains even at the temperature of 1800 degrees Fahr.

**The increase in heating time to 20 or 25 hours does not obliterate free carbides and does not appear to have much influence on relative amounts of α and γ iron.

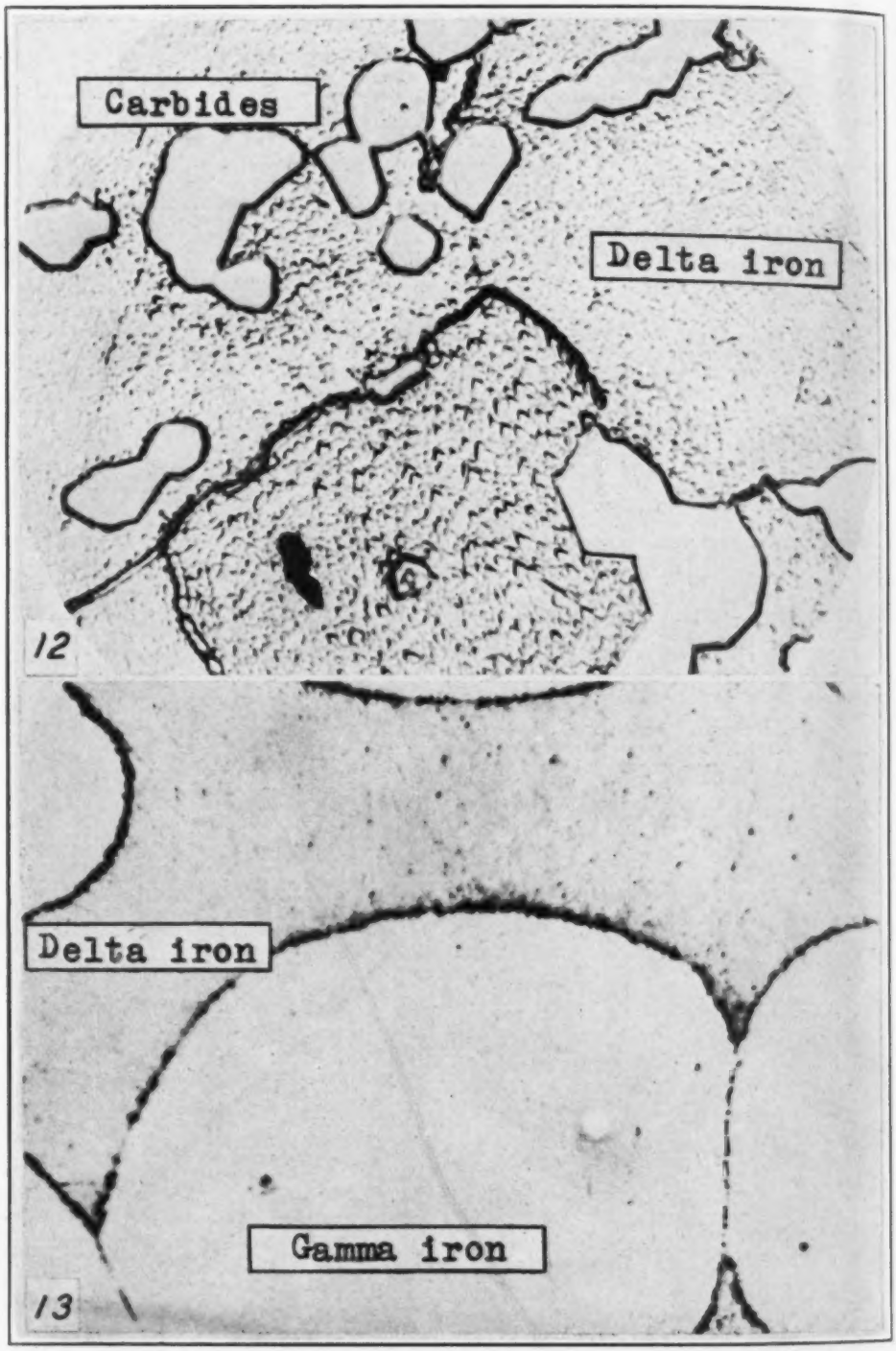


Fig. 12—Typical Delta Iron Deeply Etched with Aqua Regia, Also Some Carbides. × 2000.
Fig. 13—Typical Delta and Gamma Iron. Etched with Aqua Regia. × 2000.

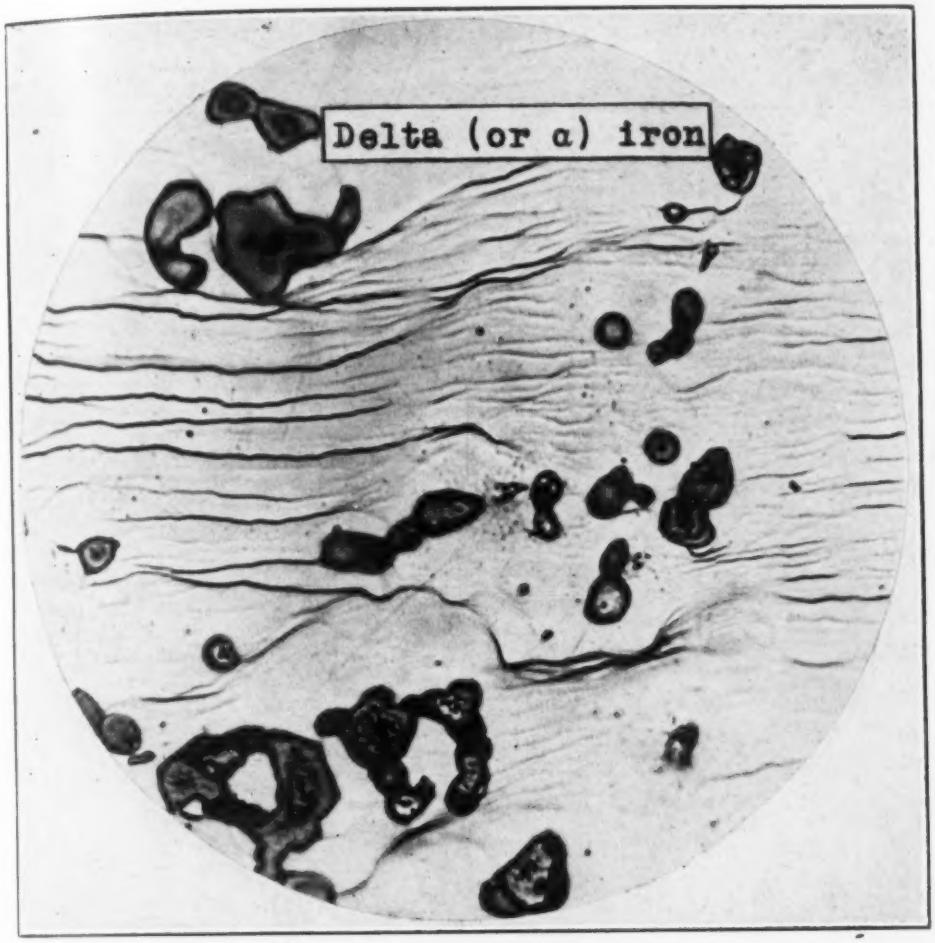


Fig. 14—Typical Appearance of Alpha or Delta Iron After Cold Working. Carbides Etched with Murakami Reagent. × 2000.

ticular chromium range the effect of variations in carbon content in relation to the temperature of heat treatment. It is assumed that the variations of the other elements is not sufficient to exert an appreciable effect on the constituents of the alloys.

The structure referred to as "grains of alpha (or delta) iron with numerous free carbides" is illustrated by the three photomicrographs, Figs. 21, 22 and 23. Incidentally, these photographs give a clear idea in regard to the procedure of studying each sample mentioned in this paper. Fig. 21 shows an unetched sample with characteristic sharply defined inclusions. The same spot is shown in Fig. 22 after 30 seconds etch with Murakami's reagent. This reagent brings out free carbides and the time of 30 seconds is quite suffi-

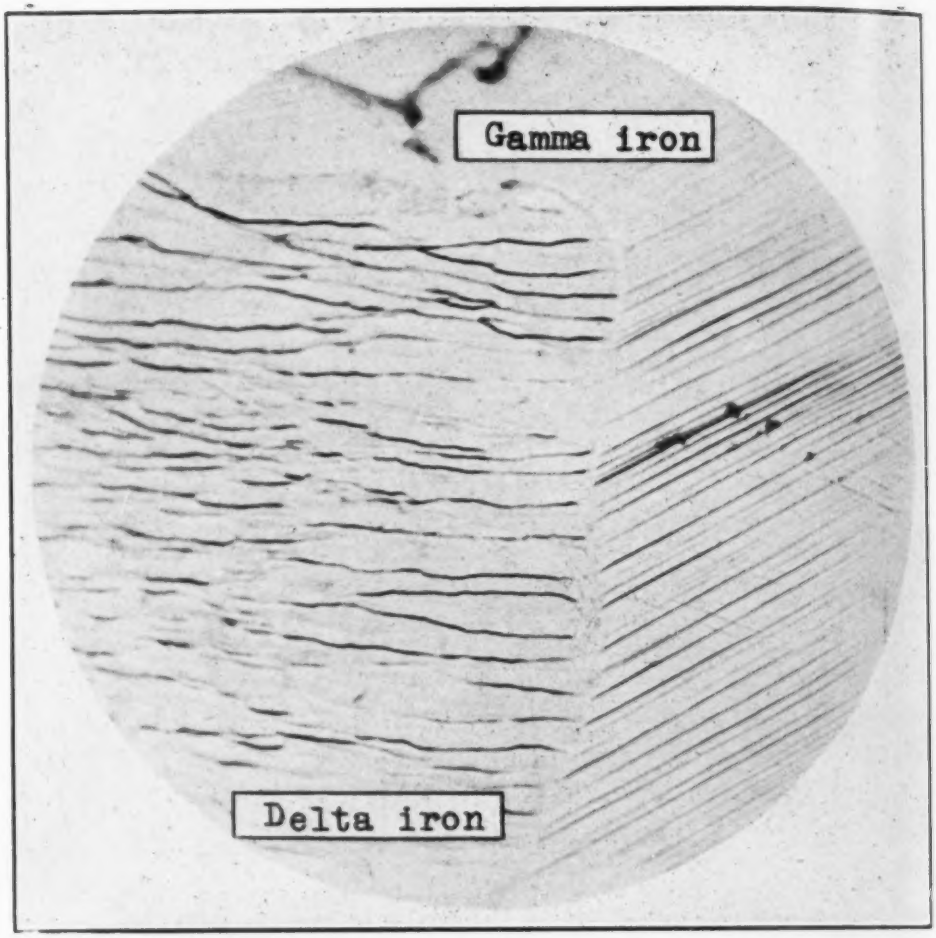


Fig. 15—Typical Appearance of Two Adjacent Grains of Alpha or Delta and Gamma Iron After Cold Working. Note Wavy Lines in Alpha Iron and Very Straight Line in Gamma Iron. Unetched. × 2000.

cient to do it. It is quite safe to assume that the amount of free carbides is negligible if the Murakami reagent fails to react within 30 seconds.

Fig. 23 shows the same spot again, this time Murakami etching being supplemented by aqua regia etch, which seems to define more sharply the already etched carbides and reveals the grain size of the sample. The structure so obtained is fully representative of the alloys, within the chemical range as given in Table I, either air-cooled from forging temperatures or quenched at relatively low heat. Of course, the size of alpha (or delta) grains as well as the fineness of carbide particles depends on the temperature and time of heat treatment. Figs. 21, 22 and 23 were taken from a sample treated for 1

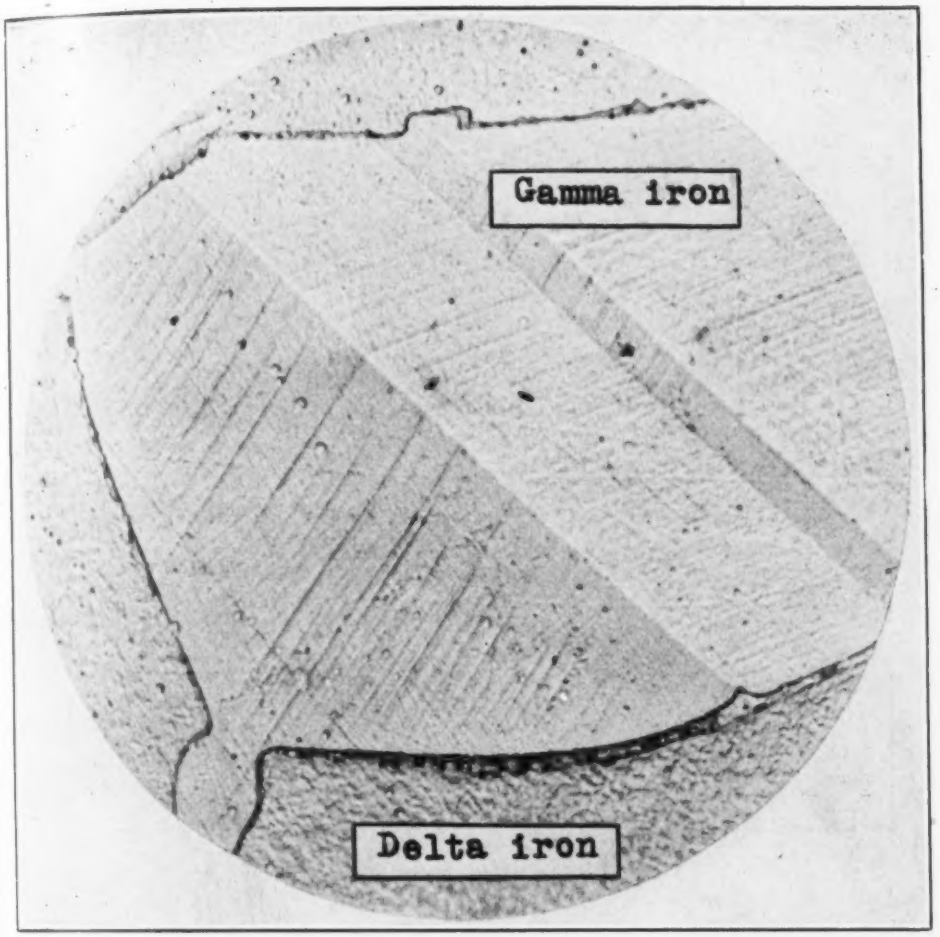


Fig. 16—Alloy No. 2235. Heat Treatment: 1 Hour at 2300 Degrees Fahr., Quenched in Ice Brine. Etched with Aqua Regia and then Cold-Worked. Twins in Gamma Iron. × 2000.

hour at 2000 degrees Fahr. The structure of the same alloy or similar alloys is much finer if treated at lower temperatures, but the structural characteristics remain the same. It must also be mentioned that the increase in carbon content has a very pronounced effect on the amount and the size of carbidic particles.

On reaching the field of higher temperature it is found that gamma iron begins to appear in limited amount, while the bulk of the previously existing alpha (or delta) structure is preserved. The temperature at which gamma iron is found is, apparently, a function of carbon content: in the alloy with 0.15 per cent carbon it is unmistakably seen first at 2100 degrees Fahr. (at 2000 degrees Fahr. the evidence is not absolutely reliable), while in an alloy with about

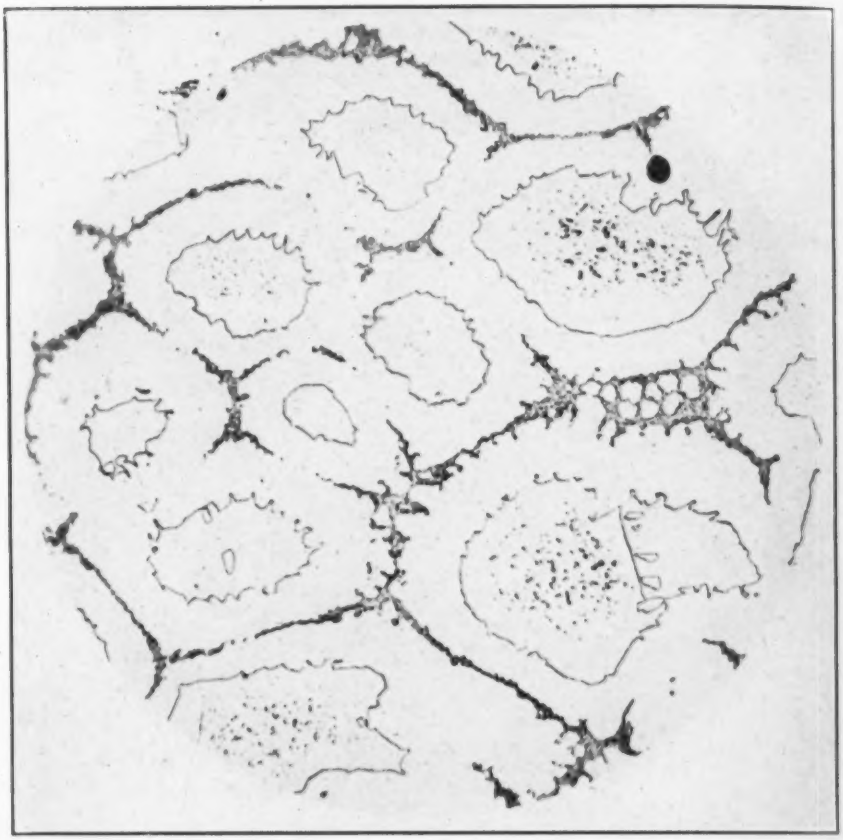


Fig. 17—Alloy No. 3535. Heated to 2500 Degrees Fahr., held 40 Minutes Quenched in Ice Brine. Etched with Murakami and Aqua Regia Reagents. Constituents: Gamma and Delta Irons, Also Eutectic. × 250.

0.20 per cent carbon, gamma iron grains are clearly seen at as low as 1900 degrees Fahr. Another alloy of the same series with 0.36 per cent carbon contains same gamma iron at 1900 degrees Fahr., which is also true of the alloy with 0.63 per cent carbon. Figs. 24 and 25 illustrate this type of structure. An interesting detail, the discussion of which must be delayed until later, is found in the observation, that when gamma iron grains appear they contain most, if not all of the free carbides, while the delta grains seem to contain none of such free carbides. The twinning in these gamma grains is not well pronounced, but, at times, can be seen through overetching. However, cold working of the sample identifies gamma iron grains beyond any doubt.

As the temperature is increased the structure shows much fewer carbides. This is true of all four alloys. No free carbides were

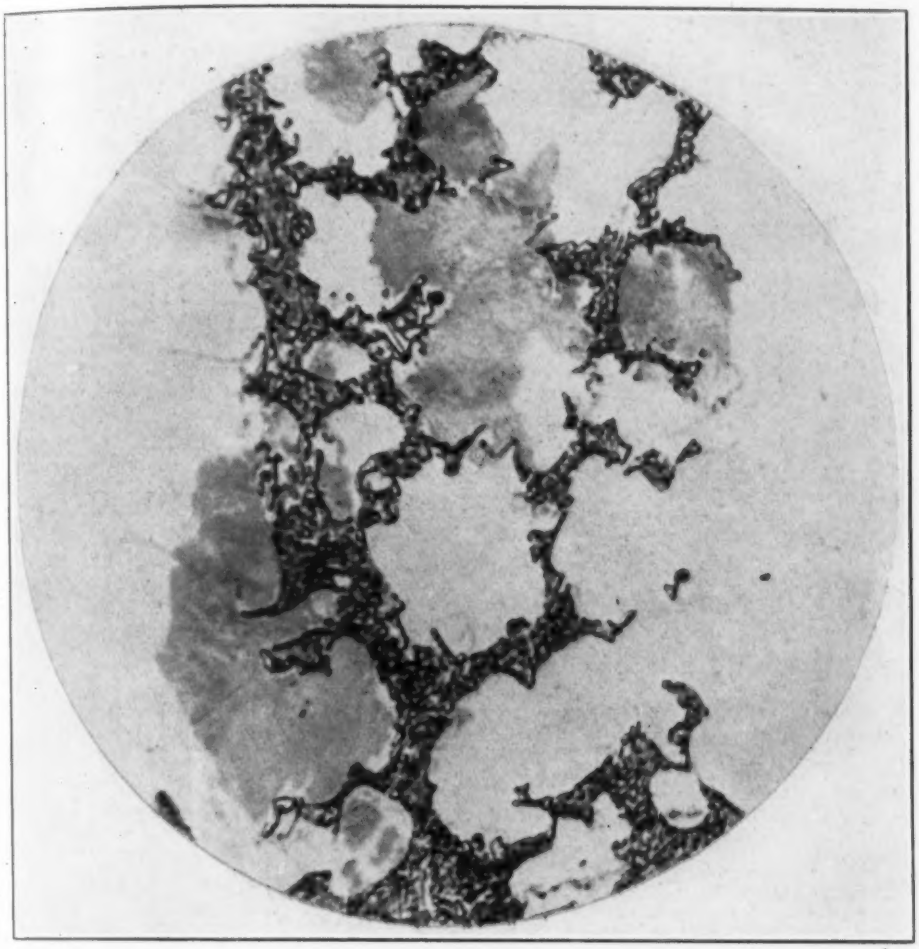


Fig. 18—Alloy No. 3535. Heated to 2500 Degrees Fahr., Held 45 Minutes, Quenched in Ice Brine. Etched with Murakami Reagent. Constituents: Gamma Iron, Eutectic and Troostite. × 2000.

found in the two alloys lower in carbon, at the temperature of 2200 degrees Fahr., while the alloy with 0.36 per cent carbon shows the absence of carbides already at 2100 degrees Fahr. It is of importance to note that the alloy No. 469 with as much carbon as 0.62 per cent contains an exceedingly large number of carbides at 2100 degrees Fahr., dispersed at random through most typical gamma grains. A powerful magnet failed to detect even traces of magnetism and it is mostly on the strength of studying this sample (as well as similar ones) that carbides are presumed to be nonmagnetic.

At 2200 degrees Fahr. the alloys containing around 0.35 per cent carbon are characterized by the structure represented in Fig. 26. Proof in regard to complete absence of free carbides is furnished by

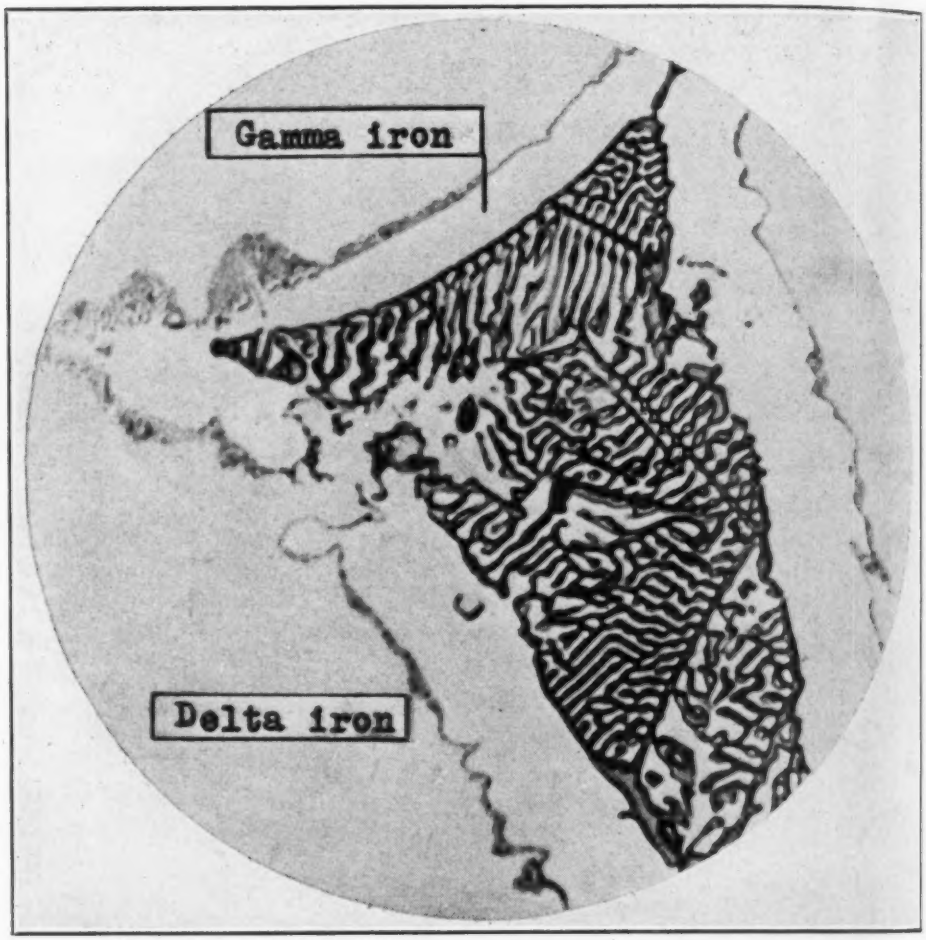


Fig. 19—Typical, Well-developed Eutectic in an Alloy Quenched from High Temperature. Etched with Aqua Regia. × 2000.

Fig. 27, representing an unetched structure of a sample and Fig. 28, showing the same spot after etching with Murakami's reagent. With a higher carbon content,—the exact amount of which would be quite unadvisable to state, carbides still are in evidence, but completely disappear at the temperature 100 degrees Fahr. higher, namely at 2300 degrees Fahr. At this temperature the structure of the alloy No. 469 (composition: Cr 21.4 per cent, C 0.62 per cent) presents a picture of pure chromium austenite alone (more or less rare in these alloys), with numerous and well-defined twinnings, (Fig. 29) the alloy being completely nonmagetic. At the same temperature (2300 degrees Fahr.) alloys sufficiently lower in carbon show the presence of delta iron, with corresponding change in magnetism,—through a feebly magnetic alloy with 0.36 per cent carbon to perceptibly mag-

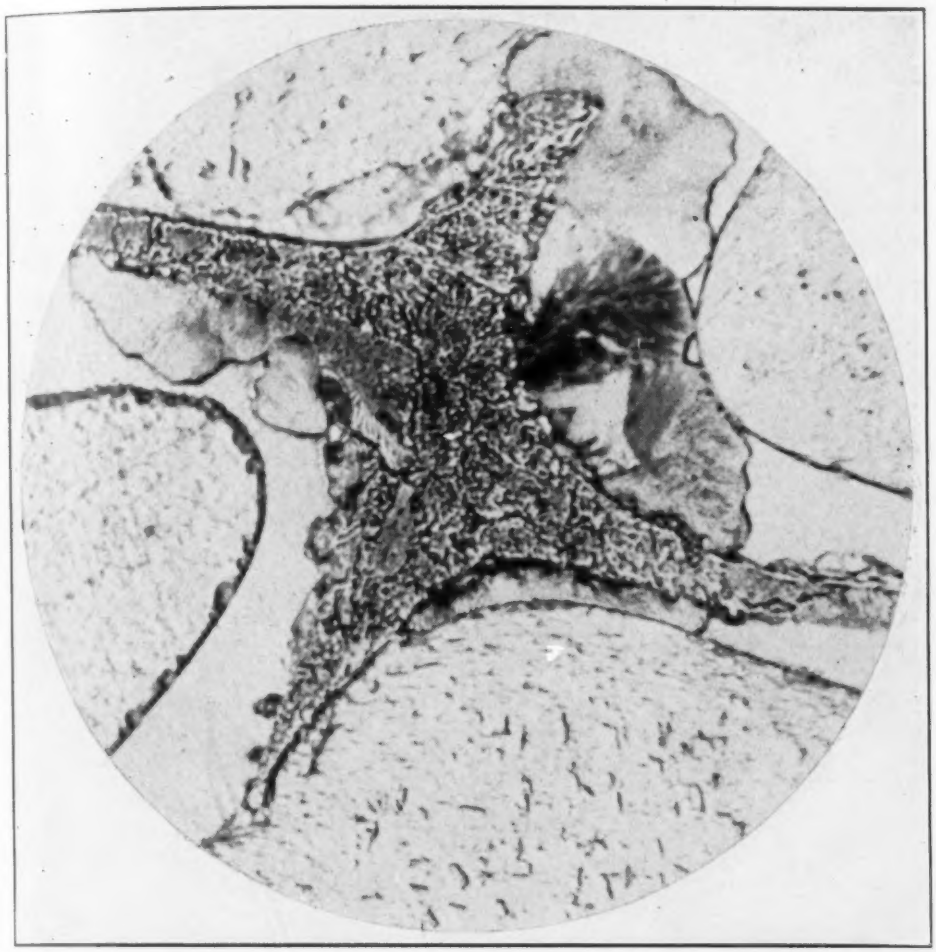


Fig. 20—Alloy No. 3535. Heated up to 2400 Degrees Fahr., Held 45 Minutes, Quenched in Ice Brine. Etched with Aqua Regia. Constituents: Gamma and Delta Irons, Some Eutectic and "Troostite-like" Constituent. × 2000.

netic with carbon around 0.20 per cent. A typical structure will be found in photomicrograph Fig. 30*, although, of course, it is somewhat modified in various alloys of this group: the modification really meaning the relative amounts of gamma and delta iron.

For the low-carbon same alloys, at the temperature of 2400 degrees Fahr. the structure remains practically the same, (Fig. 31). At higher temperatures there is a new appearance of gamma grainlets, most commonly in regular geometrical forms, within large grains of delta iron. In quite a few cases these grainlets are so distributed as to remind one of Widmanstatten patterns of cast steels, (Fig. 32). The size of delta iron grains is very large and quite often one

*The twinnings in gamma iron would be easily brought out by deeper etch.

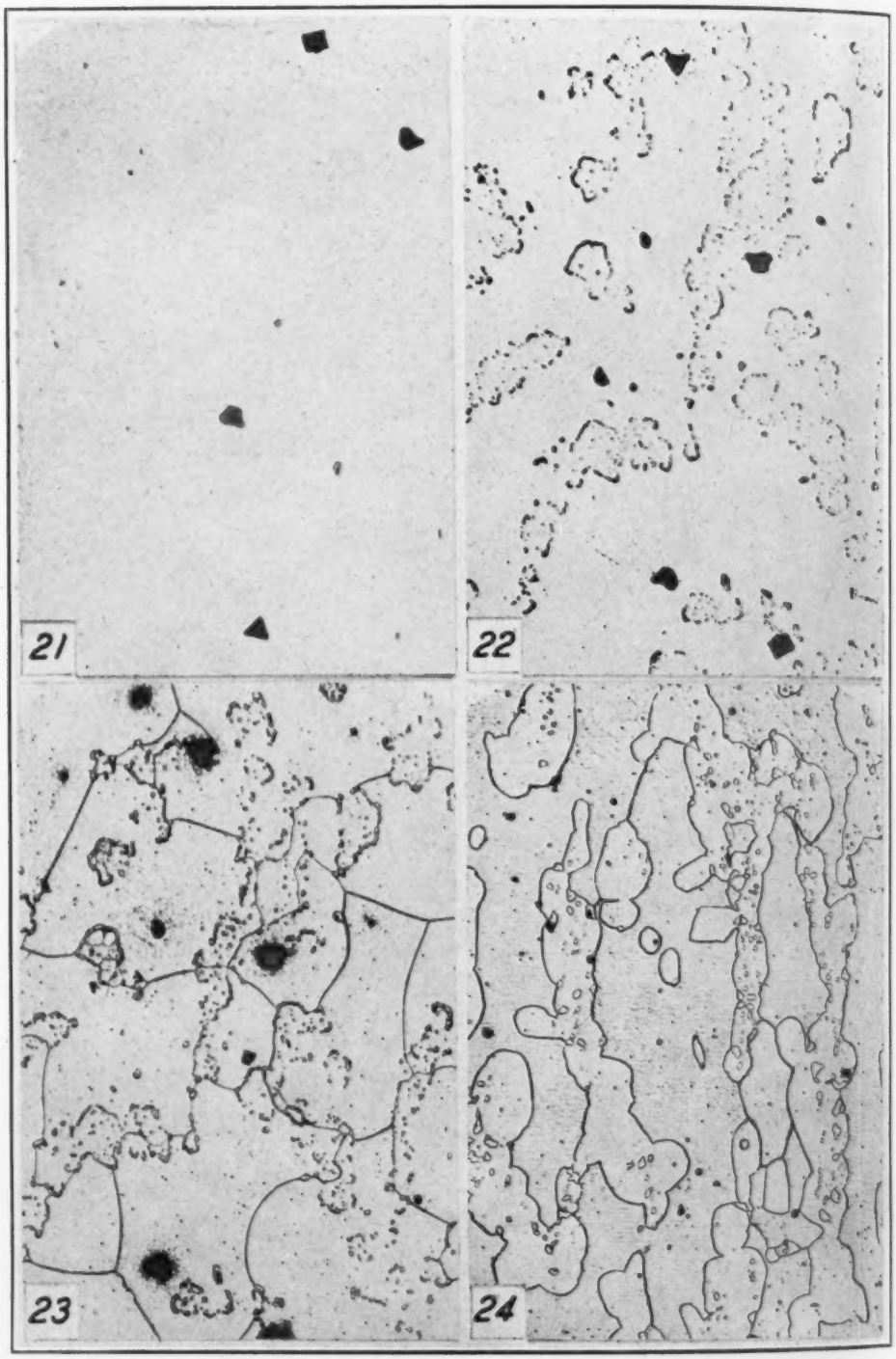


Fig. 21—Alloy 2235. Heat Treatment: 1 Hour at 2000 Degrees Fahr. Quenched in Ice Brine. Unetched. × 250.

Fig. 22—Same Sample and Same Spot as in Fig. 21. Etched with Murakami's Reagent, which Reveals the Presence of Free Carbides. × 250.

Fig. 23—Same Sample and Same Spot as in Fig. 21 and Fig. 22. Etched with Murakami and Aqua Regia Reagents. Alpha or Delta Iron and Free Carbides. × 250.

Fig. 24—Alloy 2210. Heat Treatment: 2000 Degrees Fahr., for 15 Hours. Ice Brine Quench. Delta or Alpha and Gamma Iron, also Free Carbides. Etched with Aqua Regia. × 250.

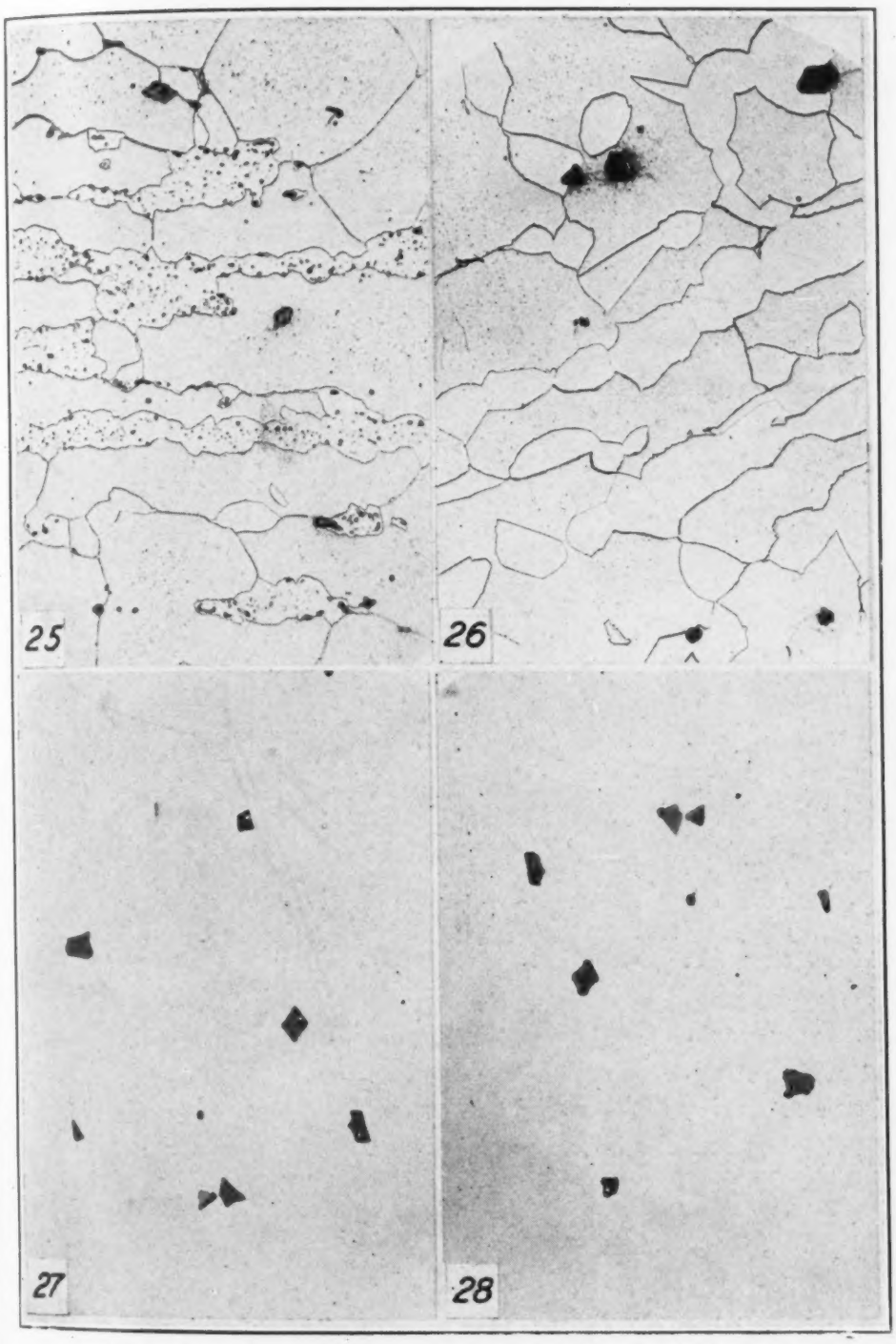


Fig. 25—Alloy 2235. Heat Treatment: 1 Hour at 2100 Degrees Fahr. Quenched in Ice Brine. Etched with Aqua Regia and Murakami Reagent. Delta and Gamma Iron, Also Free Carbides. × 250.

Fig. 26—Alloy 2235. Heat Treatment: 1 Hour at 2200 Degrees Fahr. Quenched in Ice Brine. Etched Aqua Regia. Delta and Gamma Iron. × 250.

Fig. 27—Alloy 2235. Heat Treatment: 1 Hour at 2200 Degrees Fahr. Quenched in Ice Brine. Unetched. × 250.

Fig. 28—Same Sample and Same Spot as in Fig. 27. Etched with Murakami's Reagent. All Carbides in Solution. × 250.

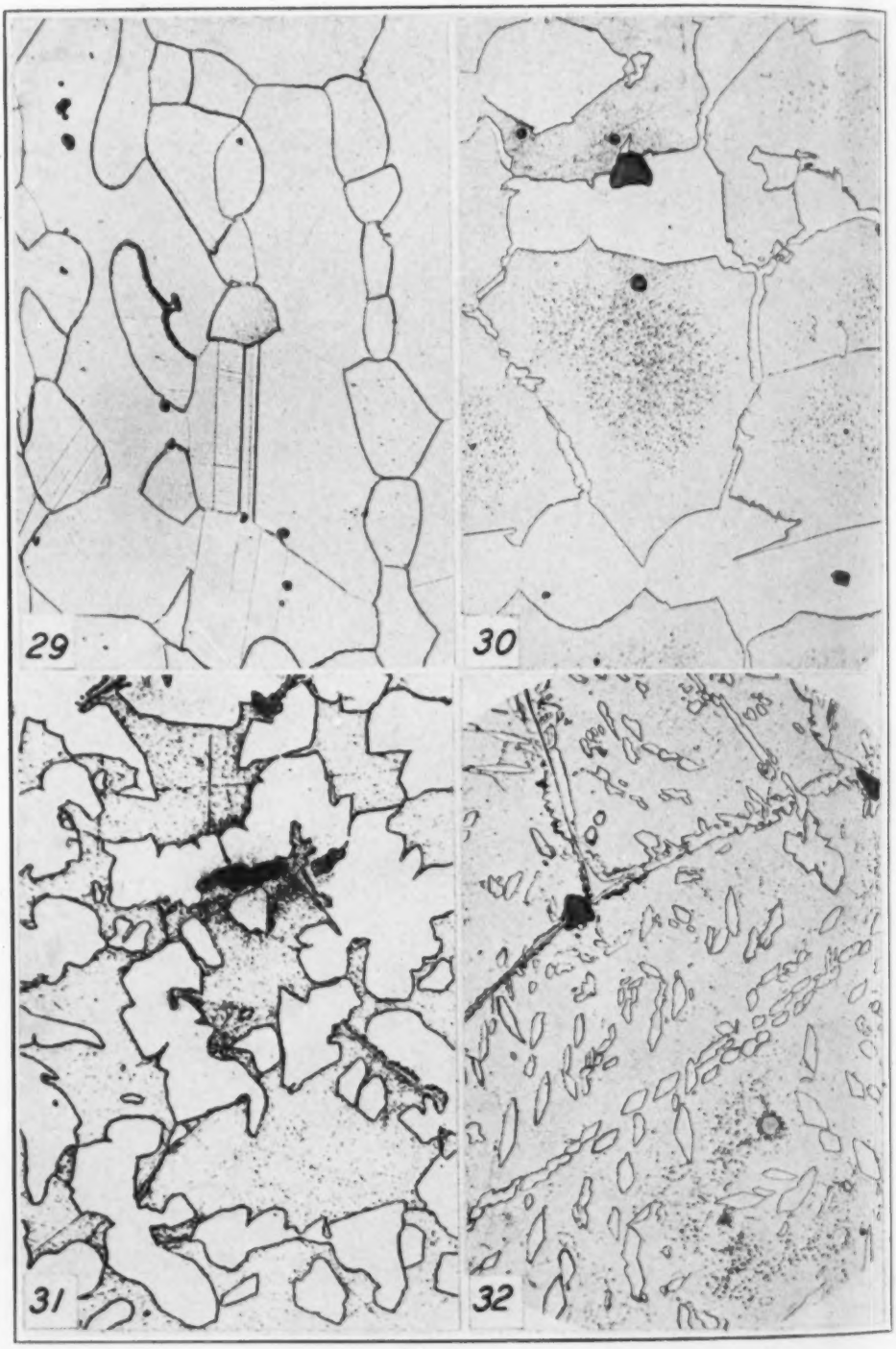


Fig. 29—Alloy 469. Heat Treatment: 2300 Degrees Fahr., for 1 Hour. Quenched in Ice Brine. Etched with Aqua Regia. Gamma Iron with Numerous Twins. Nonmagnetic. × 250.

Fig. 30—Alloy 2235. Heat Treatment: 1 Hour at 2300 Degrees Fahr., Quenched in Ice Brine. Etched with Aqua Regia. Gamma and Delta Iron in Large Grains. Free Carbides are Completely Absent. × 250.

Fig. 31—Alloy 2235. Heated to 2400 Degrees Fahr., held 30 Minutes, Quenched in Ice Brine. Constituents: Gamma and Delta Iron. Etched with Aqua Regia. × 2[illegible].

Fig. 32—Alloy 2235. Heated to 2500 Degrees Fahr., held 45 Minutes, Quenched in Ice Brine. Constituents: Delta Iron with Grainlets of Gamma Iron in Somewhat like Widmanstatten Pattern. Etched with Aqua Regia. × 250.

grain occupies the whole field of the photograph, (Fig. 33). This structure is found after quenching from 2500 degrees Fahr. It is believed that while the alloy is actually at such high temperature, the structure of it consists of but one constituent, namely delta iron, and that finding of gamma iron grainlets is due to the impossibility of quenching alloys so rapidly as to suppress altogether the delta-gamma transformation.

The other two alloys of this group containing higher carbon display different constituent at high temperatures (2400 and 2500 degrees Fahr.). These alloys remain very slightly magnetic (though practically nonmagnetic) containing, therefore, large amounts of gamma iron. A new phase—liquefied metal makes its appearance first in small amounts at grain boundaries, (Fig. 34), but at increasing temperatures and with increasing amounts of carbon it seems to spread out forming thick envelopes and also coming out within the grains (Fig. 35). No free carbides are present and it was observed that a slight increase in hardness accompanies formation of liquefied metal.

The data of constituents given in Table I can be further supplemented by the experimental results pertaining to hardness of these alloys. In the alloy No. 2235 (Fig. 36. Composition, Cr. 22.6 per cent, C 0.15 per cent) we find very low hardness after forging and slow cooling. It remains unchanged after temperatures of 1800 and 1900 degrees Fahr., is somewhat increased when the temperature of 2000 degrees Fahr. is used for quenching and attains its maximum at 2100 degrees Fahr., corresponding to the first appearance of gamma iron and a decided tendency on the part of carbides to go into solution. From 2100 degrees Fahr. on it decreases, slightly at first, but quite pronouncedly at 2300 and 2400 degrees Fahr. Presumably this change in hardness is due to the increased grain size since there appears to be no change in the constituents. Alloy No. 2210 (Fig. 36. Composition Cr. 23.7 per cent, C 0.19 per cent) hardly shows any change in hardness at all the temperatures at which hardness was measured.

Alloy 468 (Fig. 37. Composition Cr 22.0 per cent, C 0.35 per cent) indicates a much higher hardness if compared with two previous alloys: its hardness remains between 32 and 34 Rockwell C until the temperature of quenching reaches 2100 degrees Fahr. At this temperature a drop of 6 to 8 points occurs: it will be noticed that the structure at this temperature shows a complete absence of



Fig. 33—Alloy 2235. Heated to 2500 Degrees Fahr., held 45 Minutes. Quenched in Ice Brine. Constituents: Large Grains of Delta Iron and Gamma Iron Grainlets in Regular Geometrical Forms. Etched with Aqua Regia. × 250.

Fig. 34—Alloy No. 469. Heated to 2400 Degrees Fahr. Held 1 Hour. Quenched in Ice Brine. Constituents: Gamma Iron and Eutectic. Etched with Murakami Reagent and Aqua Regia. × 200.

Fig. 35—Alloy No. 469. Heated to 2400 Degrees Fahr. for 1 Hour. Quenched in Ice Brine. Gamma Iron and Thick Envelopes of Eutectic. Etched with Murakami Reagent and Aqua Regia. × 250.

free carbides. Practically no further change in hardness takes place at gradually increased quenching temperatures.

Alloy 469 (Fig. 37. Composition Cr 21.4 per cent, C 0.62 per

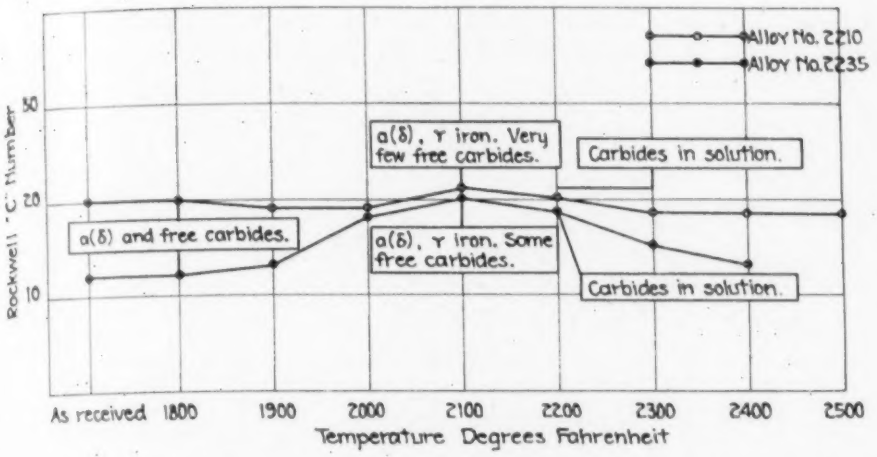


Fig. 36—Rockwell Hardness Curves and Structure of Alloys No. 2210 and No. 2235 at Temperatures Between 1800 and 2500 Degrees Fahr. The Hardness for the "As Received" Condition is also Given.

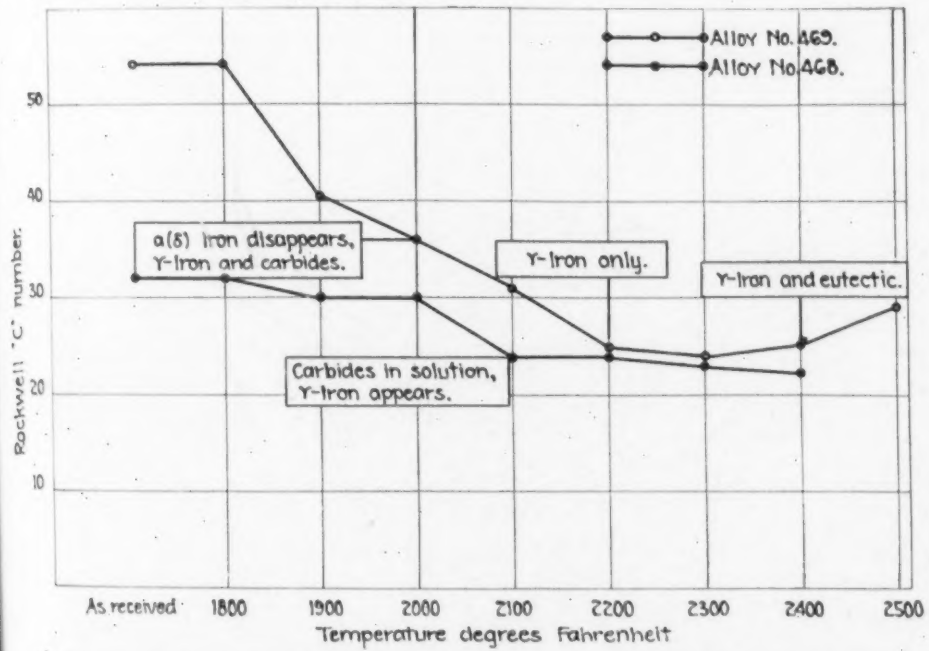


Fig. 37—Rockwell Hardness Curves and Structure of Alloys No. 468 and 469 at Temperatures Between 1800 and 2500 Degrees Fahr. The Hardness for the "As Received" Condition is also Given.

cent) shows an exceptional hardness (Rockwell C 54) in as-rolled condition. Apparently heating to 1800 degrees Fahr. and quenching produces little change since the hardness remains the same. A decided drop in hardness occurs at 1900 degrees Fahr. and again at 2000 degrees Fahr. The latter temperature corresponds to disap-

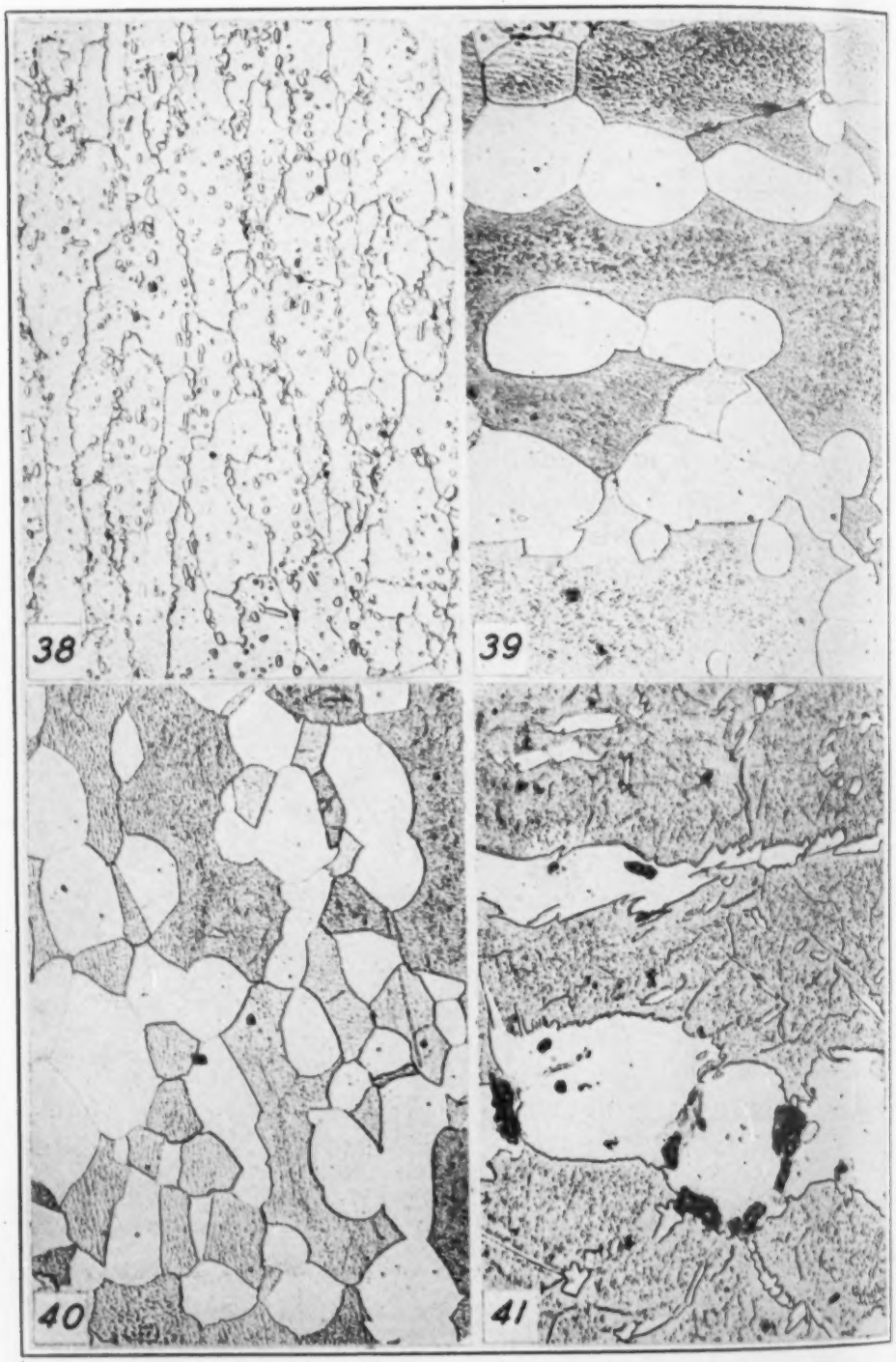


Fig. 38—Alloy 2810-1. Heated to 2000 Degrees Fahr., held 15 Hours. Quenched in water. Alpha or Delta Iron and Carbides. Etched with Aqua Regia. × 250.

Fig. 39—Alloy 2810. Heated up to 2200 Degrees Fahr. Held 4 Hours. Quenched in Ice Brine. Constituents: Delta and Gamma Iron Grains. Note Round Form of Gamma Iron Grains. Etched with Aqua Regia. × 250.

Fig. 40—Alloy 2810-6. Heated to 2300 Degrees Fahr. Held 1 Hour. Quenched in Ice Brine. Constituents: Alpha or Delta and Gamma Iron. No Carbides. Etched with Aqua Regia. × 250.

Fig. 41—Alloy 2810-8. Heated to 2400 Degrees Fahr. Held 1 Hour. Quenched in Ice Brine. Constituents: Gamma, Alpha Iron, Eutectic (?) and Troostite-like Constituent Etched with Aqua Regia. × 250.

pearance of alpha (or delta) iron phase, the structure of the alloy consisting of gamma iron grains with scattered free carbides.

At 2300 degrees Fahr. all of the carbides are driven into solution, the structure of the alloy is wholly austenitic and hardness reaches its minimum of 24-25 Rockwell C. The raising of quenching temperature to 2400 degrees Fahr. affects the hardness very little, but a certain increase was observed at 2500 degrees Fahr., at which temperature the liquefied metal appears.

Alloys with 28 Per Cent Chromium

This group contains 3 alloys (No. 2810: Cr 29.45 per cent, C 0.32 per cent; No. 2835: Cr 27.90 per cent, C 0.63 per cent; 28 CEM: Cr 29.05 per cent, C 0.45 per cent* with a fourth one (510: Cr 28.20 per cent, C 0.63 per cent) serving as a check on alloy 2835.

As far as the microscopic appearance of the constituents in alloys with 28 per cent chromium are concerned it is in many respects similar to the structures already described in some detail. The appearance of α (or δ) grains with carbides is precisely the same, as is evident from Fig. 38. The same is also true of those alloys which at elevated temperatures present the structure of α (or δ) and γ grains. One interesting detail deserves attention: γ iron grains appear to be of the round form, in certain cases being almost perfect circles (Fig. 39). Several experiments were performed with the idea in view that much longer time of heating at certain temperatures might perceptibly change the constituents characteristic of these temperatures, or at any rate, modify the appearance and distribution of constituents. However, such was not the case: Alloy 2810 displays the same structure of δ (or α) iron with numerous carbides, whether it was held at, for example, 2000 degrees Fahr. for 1 hour or for 15 and even 30 hours (Fig. 42). Of course there is a perceptible difference in grain size but all other characteristics remain the same and there is no evidence whatsoever that the carbides tend to go into solution, no matter how prolonged the heating at the mentioned temperature may be. The same is equally true of other alloys and of other temperatures: Alloy No. 2835 may be mentioned the structure of which consists of α (or δ) iron and carbides whether it was maintained at 2000 degrees Fahr. for 1 hour or for 15 hours; again maintaining the same alloy at 2300 degrees Fahr. a structure

*Alloy 28 CEM contains also 0.98 per cent Nickel.

Table II

Alloys with 28 Per Cent Chromium

Summary of the Constituents at Different Temperatures

Temperature	Carbon Content Per Cent 0.32	0.45	0.63	Check on 0.60 (0.63-0.65)
2500° F. 1370° C.	This alloy is in pasty condition at this temperature. The constituents are: large grains of δ iron with smaller γ grains. δ iron predominates. Only traces of liquefied metal here and there, in isolated patches—not at the grain boundaries. Magnetic.	Somewhat pasty at this temperature. δ iron grains surrounded by liquefied metal. The latter is, in turn, surrounded by γ iron, but only in places. Individual patches of eutectic within δ grains. Strongly magnetic.	Somewhat pasty at this temperature. Very well developed liquefied metal, surrounded by some γ iron. Large δ iron grains. Magnetic.	Somewhat pasty at this temperature. Very pronounced liquefied metal at the grain boundaries and within δ grains. Magnetic.
2400° F. 1315° C.	The structure is practically the same as at 2500° F. No liquefied metal. Some small γ grainlets are seen within large δ grains. Magnetic. Fig. 41.	δ iron grains, surrounded by liquefied metal. Large amount γ iron grainlets and eutectic formations within δ grains. Fig. 43.	δ and γ iron in about equal proportions. Liquefied metal at the grain boundaries. No separation of γ iron around eutectic. Slightly magnetic.	Very pronounced liquefied metal at the grain boundaries and within the grains, surrounded by γ iron. Large δ iron. No free carbides (except as part of the liquefied metal.) Slightly magnetic. Figs. 44 and 45.
2300° F. 1260° C.	δ and γ grains in about equal amounts. On deep etching γ iron grains show distinct twinings. Magnetic. Fig. 40.	δ (or α) and γ grains, the former somewhat predominating. Abundance of free carbides. Magnetic.	δ and γ iron grains in about equal amounts. An appreciable amount of free carbides. Slightly magnetic.	δ and γ iron grains in about equal amounts. Large amount of free carbides. Slightly magnetic.
2200° F. 1205° C.	Larger δ grains, smaller γ grains containing some free carbides. γ grains display evident twinning. Magnetic. Fig. 39.	δ (or α) and γ grains, the former decidedly predominating. Abundance of free carbides. On cold working γ iron shows beautiful twins. Magnetic.	δ (or α) grains almost entirely. Only a few γ iron. An appreciable amount of free carbides. Magnetic.	δ (or α) iron grains with only a very few γ grains, seen after cold work. Very large amount of carbides. Magnetic.
2100° F. 1150° C.	Mostly δ (or α) grains with numerous carbides. A few γ grains. Magnetic.	δ (or α) iron. Plentiful carbides. Magnetic.	δ (or α) iron grains medium size. Numerous carbides. Magnetic.	Fairly large grains of δ (or α) iron. Numerous free carbides. Magnetic.
2000° F. 1095° C.	δ (or α) grains, fairly large in size, with very numerous carbides. Magnetic. Fig. 38.	δ (or α) iron. Plentiful carbides. Magnetic.	δ (or α) iron grains medium size. Numerous carbides. Magnetic.	δ (or α) grains with numerous carbides. Magnetic.
1900° F. 1040° C.	δ (or α) grains with very numerous carbides. Magnetic.	δ (or α) iron. Plentiful carbides. Magnetic.	δ (or α) iron grains medium size. Numerous carbides. Magnetic.	δ (or α) grains with numerous carbides. Magnetic.
1800° F. 985° C.	δ (or α) grains with very numerous carbides. Magnetic.	δ (or α) iron. Plentiful carbides. Magnetic.	δ (or α) iron grains medium size. Numerous carbides. Magnetic.	δ (or α) grains with numerous carbides. Magnetic.
Alloy No.	2810	28 CEM	2835	510

of δ and γ iron plus free carbides is found regardless of the duration of heating time. This last observation is somewhat surprising for it indicates that the equilibrium calls for all three phases, α iron, γ iron and carbide.

Within the range of temperatures between 2400-2500 degrees Fahr. the structure of the alloy with 0.32 per cent carbon is quite the same as found in 22 per cent chromium alloys, and the photomicrograph of the latter (see Fig. 31) could represent an alloy with 28 per cent chromium. The structure of a 28 per cent chromium alloy, however, contains in addition to δ grains with γ grainlets quite a perceptible number of fairly large γ iron grains, more or less equally distributed among δ iron. δ iron grains appear to contain much lesser amount of γ grainlets than in case of 22 per cent chromium alloys and this observation, repeatedly made, allows the suggestion that with the increase in chromium content δ iron becomes somewhat more stable. Another difference in the structure of 22 and 28 per cent chromium alloys at high temperatures is that the latter contain evident traces of formation of liquefied metal, not in the continuous patterns but in isolated patches.

With increase in carbon content to 0.43 per cent the structures of the alloy at the temperatures up to 2300 degrees Fahr. does not show any exceptional features and are fully covered by descriptions contained in the Table II. At higher temperature (2350-2400 degrees Fahr.) it is found that this alloy shows the presence of γ grainlets in large δ grains although some of γ iron is found as part of the formed eutectic as is illustrated in Fig. 43. When quenching of this alloy is accomplished at the temperature of 2500 degrees Fahr., or as was tried even higher (although the alloy softened quite perceptibly—the structure is again made up of δ grains, surrounded by eutectic, but the number of γ iron grainlets is decidedly diminished. Quite apparently higher temperature of quenching precludes the separation of γ iron constituent. For the alloys with 0.60 per cent carbon a characteristic structure at 2400 degrees Fahr. is represented by Fig. 44, showing large δ iron grains with the eutectic for grain boundaries, the eutectic, in turn, being surrounded by fairly thick envelopes of γ iron. Most of the δ grains contain also quite a large amount of individual eutectic formations (Fig. 45) and from the comprehensive and comparative study of numerous different alloys it becomes evident that the amount of the eutectic increases with the in-

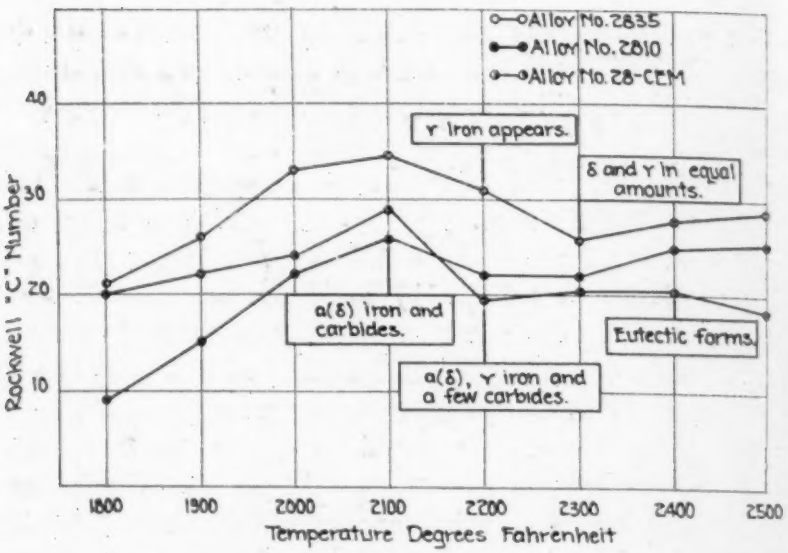


Fig. 42—Rockwell Hardness Curves and Structure of Alloys No. 28-CEM, No. 2810 and No. 2835 at Temperatures Between 1800 and 2500 Degrees Fahr.

Table III
Alloys with 33 to 34 Per Cent Chromium
Summary of the Constituents at Different Temperatures

	Carbon Content Per Cent	
Temperature	0.25-0.27	0.60-0.62
2500° F.	Very large grains of δ iron with a few γ iron grainlets. Very poorly developed liquefied metal and "troostite" in massive formations around grains. Magnetic.	Large grains of δ iron, surrounded by γ iron and eutectic. Magnetic. Figs. 17 and 18.
2400° F.	Fairly large grains of δ and γ iron with occasional "troostitic" formations. Magnetic.	Fairly large grains of δ and γ iron, surrounded by the liquified metal in the first stages of formation. Magnetic. Figs. 20 and 50.
2300° F.	Grains of δ and γ iron with numerous "troostitic" formations. Magnetic.	Grains mostly of δ with some γ iron with "troostitic" formations. Free carbides. Magnetic. Figs. 48 and 49.
2200° F.	Grains of δ and γ iron, free carbides and "troostitic" formations. Magnetic.	Grains of δ and γ iron, free carbides and "troostitic" formations. Magnetic. Fig. 47.
2100° F.	Grains of δ (or α) iron and very numerous free carbides. This structure is maintained all way down to room temperature, the only perceptible difference being in the grain size of δ (α) grains. Magnetic.	Grains of δ (or α) iron with numerous carbides. The structure does not change with the lowering of the temperature. Magnetic.
Alloy No.	3510	3535

crease in carbon and chromium contents.

Alloys with 33 to 34 Per Cent Chromium

As was done previously the summary concerning the constituents of alloys with 33-34 per cent chromium is given in Table III.

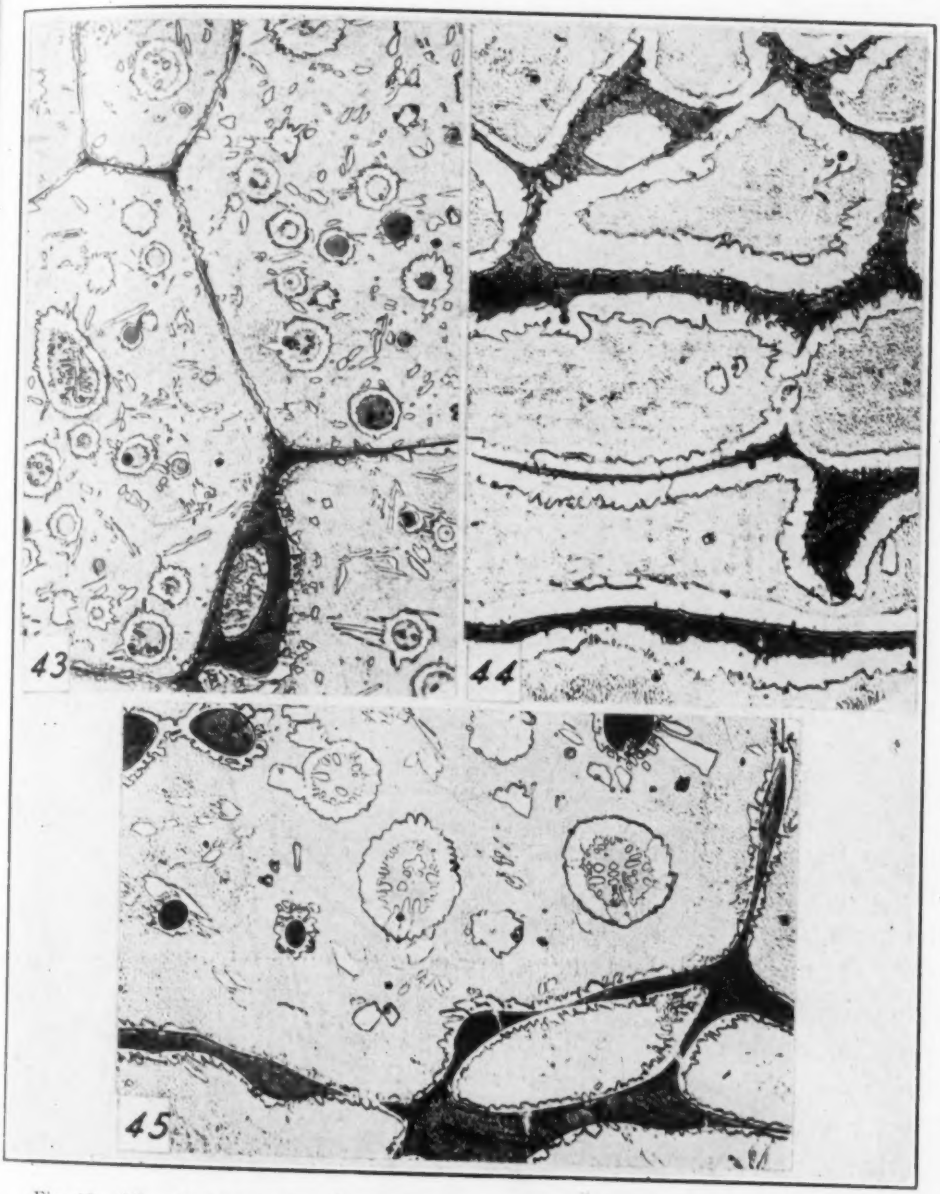


Fig. 43—Alloy 28-CEM. Quenched from 2400 Degrees Fahr. in Ice Brine. Constituents: Delta Iron Grains with Numerous Gamma Iron Grainlets, Sometimes in Widmanstatten Patterns. Liquefied Metal at Grain Boundaries and to Some Extent within Delta Grains. Etched with Aqua Regia. × 250.

Fig. 44—Alloy 510. Quenched in Ice Brine from 2400 Degrees Fahr. Constituents: Delta Iron Grains, Liquefied Metal Surrounded by Gamma Iron. Etched Aqua Regia. × 250.

Fig. 45—Alloy 510. Quenched in Ice Brine from 2400 Degrees Fahr. Constituents: Delta Iron Grains with Gamma Iron Grainlets and Numerous Liquefied Metal Formations. Liquefied Metal at Grain Boundaries. Etched with Aqua Regia. × 250.

As in previous cases the structure of these alloys at relatively low temperatures does not require special discussion: in all cases in which

δ (or α) iron with numerous carbides was found its structural characteristics are precisely the same as in the alloys of different composition. It is only when we enter the field of higher temperatures that either a new constituent appears or a familiar (from the previous discussion) one takes on somewhat different appearance.

In this series of alloys the "troostitic" constituent, already referred to, is most conspicuously pronounced. This constituent has

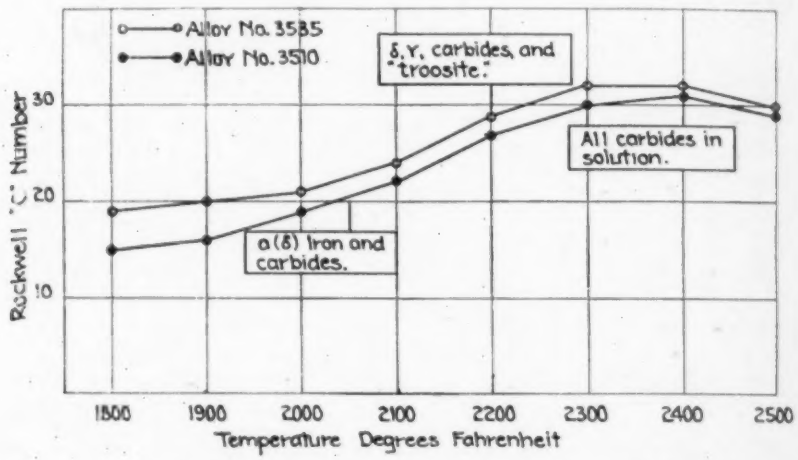


Fig. 46—Rockwell Hardness Curves and Structure of Alloys No. 3510 and No. 3535 at Temperatures Between 1800 and 2500 Degrees Fahr.

never been found at temperatures lower than 2200 degrees Fahr., but when found its relative amount does not seem to depend on the temperature: it is quite true of both alloys No. 3510 and 3535 (Fig. 46). It might well be that its presence depends entirely on the rate of cooling and on the solubility of carbides in chromium γ iron. The metallographic characteristics of "troostitic" formations do not yield information to establish the nature of this constituent. In all its behavior toward different etching reagents a "troostitic" grain is apparently much the same as is its prototype in plain carbon steels. Consequently, its structural constituents are admitted to be magnetic iron and iron-chromium carbides, apparently in a very fine degree of dispersion. Such an assumption seems not illogical and is supported, as was already mentioned, by the methods of metallographic studies.

However, the reasons for "troostitic" formations are not ascertained as easily.

Certain interesting observations relative to troostitic grains have been made and deserve attention. Troostitic grains are, in the overwhelming majority of cases, located at the boundary line between

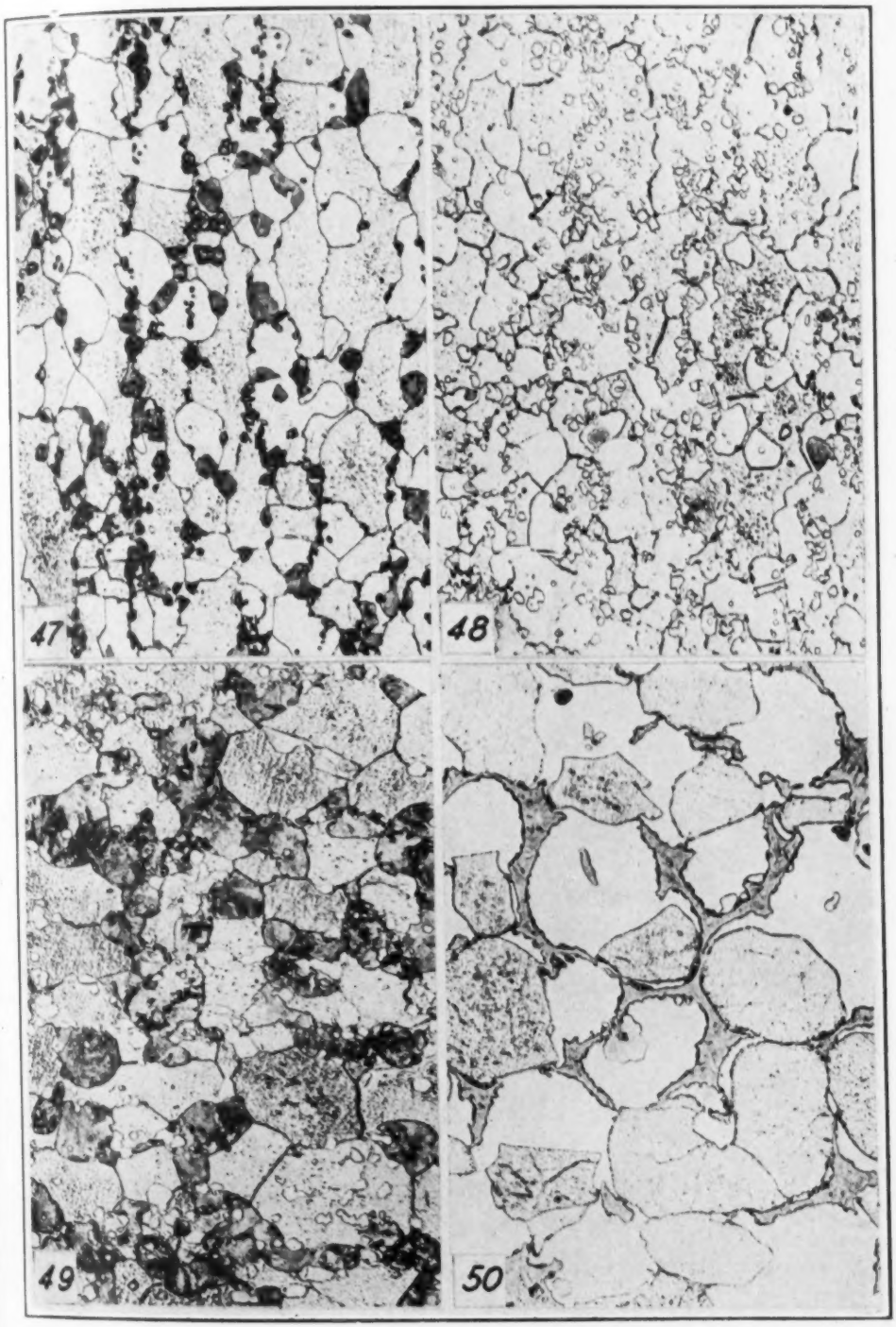


Fig. 47—Alloy 3535. Heated up to 2200 Degrees Fahr. for 4 Hours, Quenched in Ice Brine. Constituents: Delta and Gamma Iron Grains, also Troostite. Etched with Aqua Regia and Murakami Reagent. × 250.

Fig. 48—Alloy 3535. Heated to 2300 Degrees Fahr. for 1 Hour, Quenched in Ice Brine. Constituents: Delta and Gamma Iron Grains, also Troostite. Etched with Aqua Regia and Murakami Reagent. × 250.

Fig. 49—Alloy 3535. Same Sample as in Fig. 48. Heated up to 2300 Degrees Fahr. for 15 Hours, Quenched in Ice Brine. Constituents: Delta and Gamma Iron Grains. Note Increase in Troostite Formations. Etched with Aqua Regia and Murakami Reagent. × 250.

Fig. 50—Alloy 3535. Heated to 2400 Degrees Fahr. for 1 Hour, Quenched in Ice Brine. Constituents: Delta and Gamma Iron Grains, Liquefied Metal and a Few Troostitic Formations. Etched with Aqua Regia. × 250.

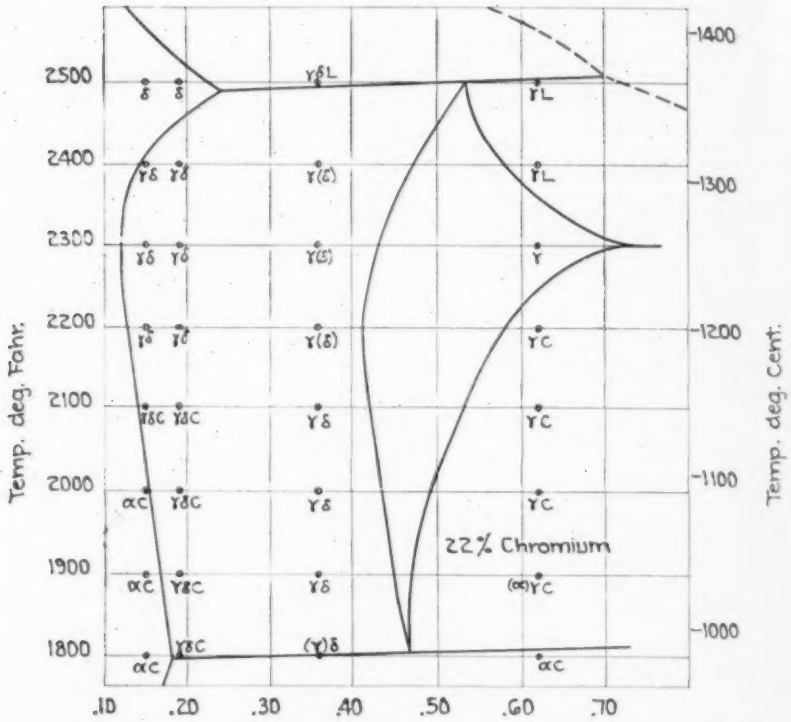


Fig. 51—The Structures Found in Alloys with 22 Per Cent Chromium.

δ and γ iron grains when the latter two are present simultaneously. It seems, further, that the enlargement of troostitic formations is accomplished by the invasion of γ iron areas through, evidently, some process of decomposition of γ solid solution. A photomicrograph illustrating this assertion will be found in Fig. 47. It may be entertained that the underlying cause of troostitic growth is due to the solubility phenomenon, much of the same nature as has been discovered in the γ solid solution of high manganese steels.* It might again be mentioned that in the presence of δ and γ iron any free carbides that may be present have, almost invariably, been found within the grains of γ chromium iron. It is believed that this fact and the invasion of γ iron areas by troostitic growth are undoubtedly related.

The relative amount of troostitic formations seems to be influenced by the length of time at high temperatures. A comparison of Fig. 48 showing only a few troostite-like grains after 1 hour at 2300 degrees Fahr. and Fig. 49 showing the structure of the same alloy (No. 3535) after 15 hours at the same temperature is of interest.

*V. N. Krivobok, "A Study on the Constitution of High Manganese Steels," TRANSACTIONS, American Society for Steel Treating, June, 1929. Vol. XV, p. 893.

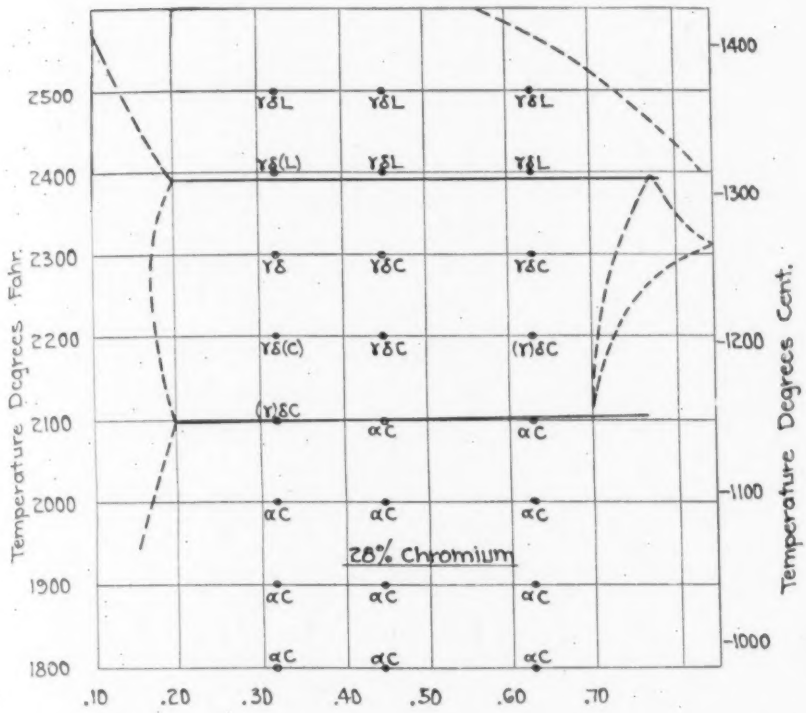


Fig. 52—The Structures Found in Alloys with 28 Per Cent Chromium.

The structural characteristics of eutectic appearing in the alloy No. 3535 at 2400 and 2500 degrees Fahr. are somewhat different. In the first place the liquefied metal formed at 2400 degrees Fahr. and for that matter at 2450 degrees Fahr., is not surrounded by γ iron (Fig. 50) while such is invariably the case with the liquefied metal formed at 2500 degrees Fahr. (Fig. 17). Secondly, the former is also quite often accompanied by a few troostitic grains and the similarity between the two is so great that the only criterion for the discrimination is the flower-like shape of troostitic growth.

Turning to a careful study of the curves of hardness (Figs. 42 and 46) an interesting observation becomes at once apparent: the hardness of 28 and 33 per cent chromium alloys (regardless of carbon content) is on the increase with raising of temperature of quench. In all three alloys with 28 per cent chromium the maximum in hardness lies somewhere between 2100 and 2200 degrees Fahr., while in 33 per cent chromium the maximum hardness is shifted to approximately 2300 degrees Fahr. It might also be recalled that 22 per cent chromium alloy with carbon up to 0.20 per cent [i. e. with α (δ) iron

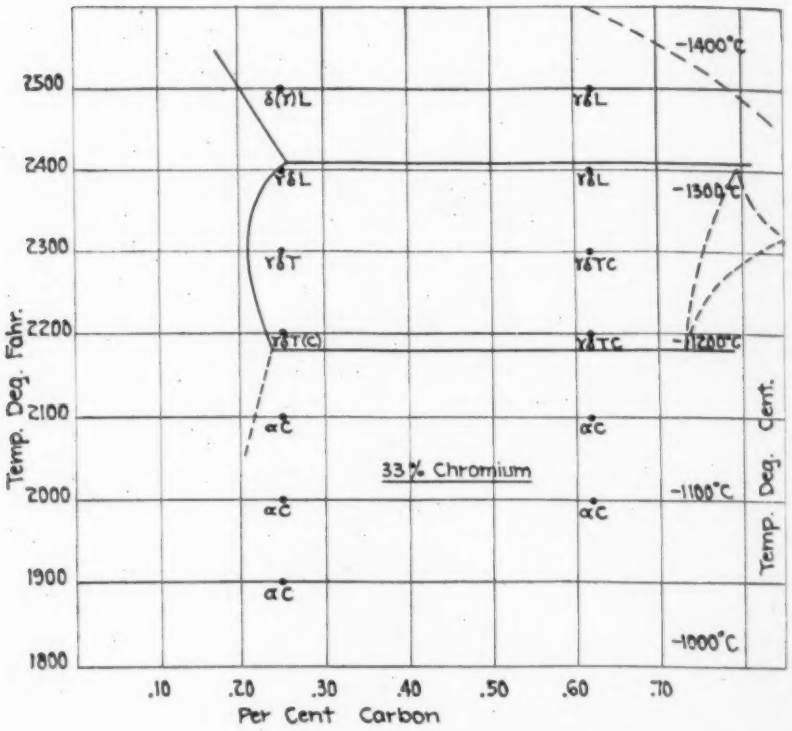


Fig. 53—The Structures Found in Alloys with 33 Per Cent Chromium.

and carbides persisting to a relatively high temperature] shows the same general type of hardness curve (Fig. 36).

Metallographic studies show that starting with the quenching temperature of 1800 degrees Fahr. and gradually increasing it the same type of structure [α (δ) iron and carbides] is preserved with carbides gradually decreasing in size and number, obviously going into solution. Parallel with this, as has been shown, the hardness of the alloy increases and in certain cases (for instance in alloys 3510 and 3535—Fig. 46) it actually reaches maximum hardness when *all* of the carbides are in solution. Apparently, solution of carbides has a very definite strengthening influence on iron of these alloys so much so that it overcomes the increase in grain size due to the higher temperature of quenching and also the strengthening afforded by the presence of hard carbidic particles.

Conclusion

The structures obtained at various temperatures in the different alloys can be related quite simply in a ternary constitutional diagram.

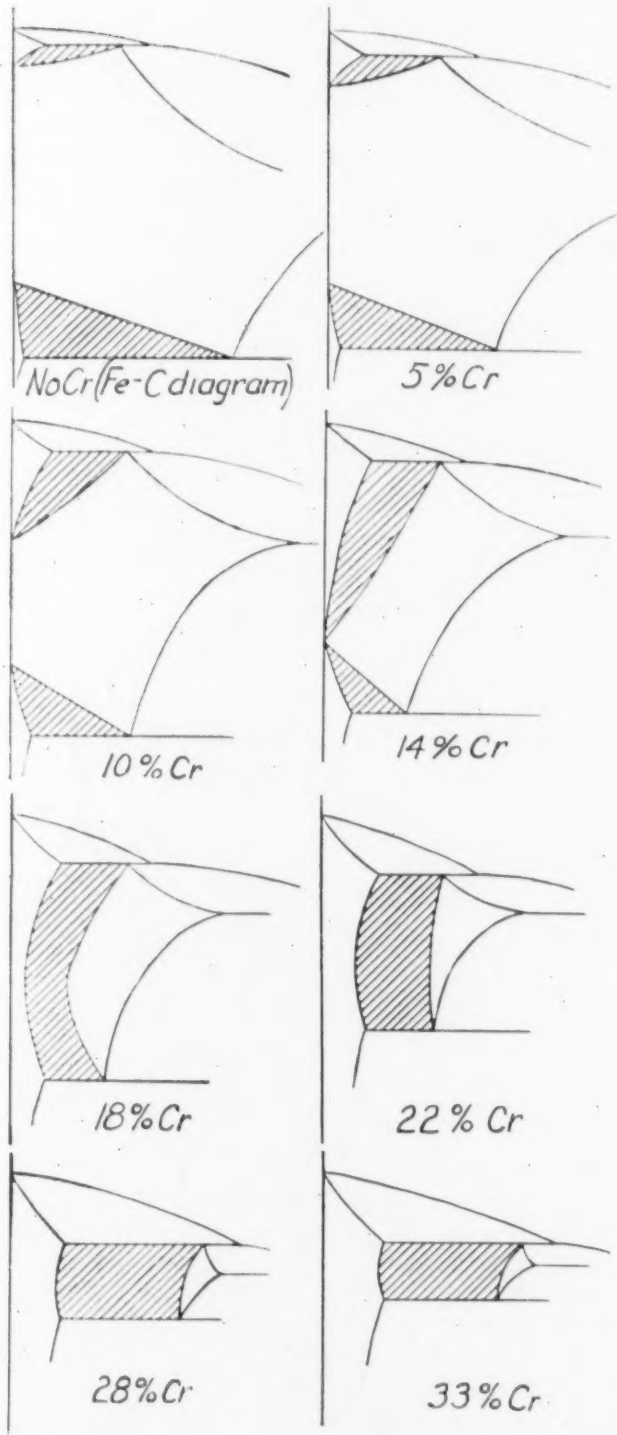


Fig. 54—Sections of Ternary Constitutional Diagram at Successively Higher Percentage of Chromium.

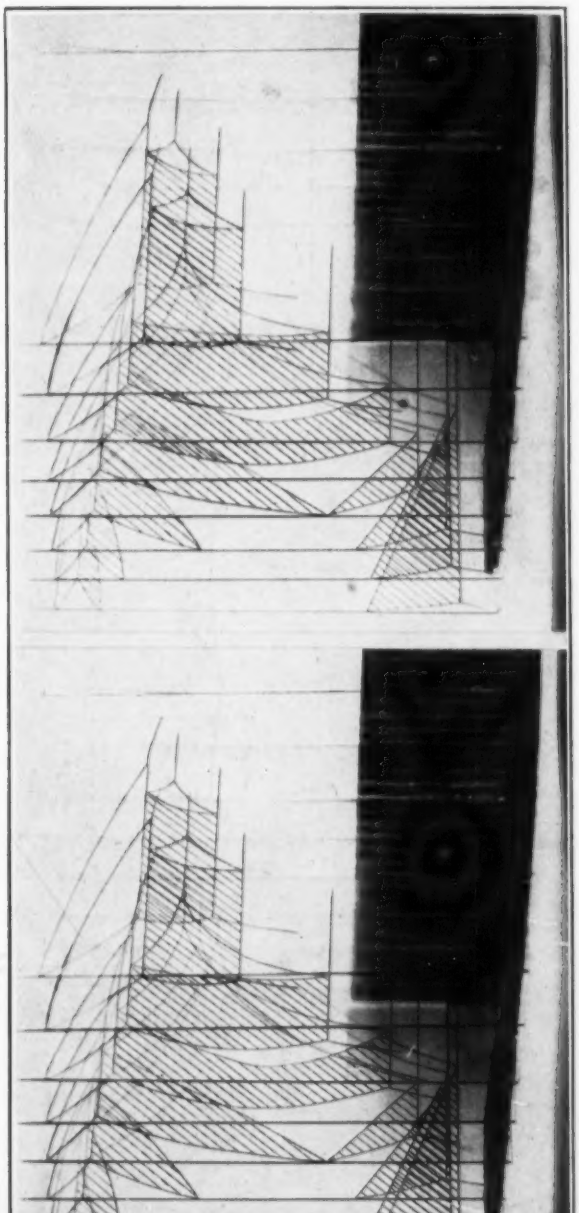


Fig. 55.—Section of Ternary Diagram to be Viewed in a Stereoscope.

This was pointed out at the beginning of the present paper, and it was stated that these relationships would be indicated by considering cross-sections of the ternary diagram.

Figs. 8 and 9 show how, with increasing chromium content, the familiar fields of the iron-carbon diagram change their configura-

tion. Those diagrams, together with Figs. 6 and 7, trace the changes up to 18 per cent chromium. It will now be seen how these regions change still further when the chromium content undergoes a considerable increase.

Tables I, II and III list the structures obtaining in three sets of alloys, one set at 22 per cent chromium, one at 28 per cent chromium, and one at 33 per cent chromium. These structures can be plotted on charts in which the abscissae are carbon contents and the ordinates are temperatures (as in an iron-carbon diagram). This is done in Figs. 51, 52 and 53. It is found that the structures may properly be grouped in fields, and that these fields form a regular progression with the fields discussed previously. In each case there is a distinct range of carbon content and temperature in which iron occurs alone, where it occurs with carbides, and another where it is associated with austenite. There are also the liquidus and the field of pure austenite.

ACKNOWLEDGMENTS

The authors wish to express their indebtedness, especially to Dr. J. Friauf for X-ray spectrometry, to Dr. Charles Austin of the National Tube Co. for the use of equipment, to Miss Z. Hill and G. Martin for assistance in preparation of specimens, to K. B. Bowman for the many chemical analyses, to E. R. Johnson and J. C. Joublanc for preparation of melts of steel, and to Dr. R. H. Aborn for X-ray spectrometry.

Addendum I

Thermal Analysis of Iron-Chromium-Carbon Alloys

BY V. N. KRIVOBOK

The study of the constituents of iron-chromium-carbon alloys as related in the present paper has been further advanced through the application of thermal analysis. In view of the nature of some of the phases (such as eutectic) and because of the high temperature at which such phases occur, the investigation necessitated special arrangements, such as a furnace, for example, capable of attaining high temperatures at a uniform rate.

The furnace was that of a small size tubular type, "globar" heated, the samples, with the thermocouple inserted in a drilled out hole, being placed in a smaller tube closed from both ends. The rate of cooling and heating could be maintained quite steady, as is seen from the introduced copies of original critical curves. Readings were taken every 0.025 millivolts (corresponding to about 2 degrees Cent.) by means of type K potentiometer and, when obtained, plotted to give an inverse rate cooling or heating curve.

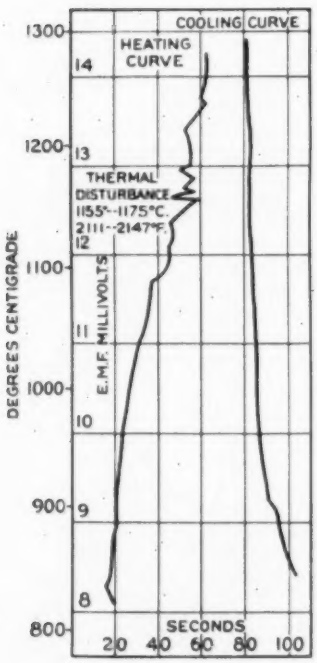


Fig. 1

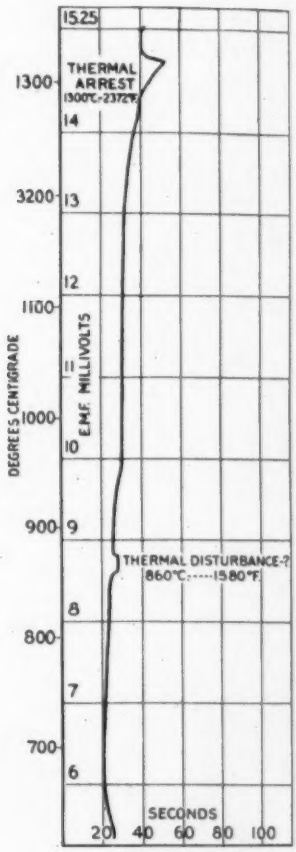


Fig. 2

Every alloy mentioned in the paper was so analyzed. However, only curves representing four alloys, typical for given series, are reproduced.

Fig. 1 shows heating and cooling curves for an alloy No. 468 with 20-22 per cent chromium and 0.35 per cent carbon. This curve shows no thermal disturbance, whatsoever, on heating from room temperature up to about 850 degrees Cent., at which a very slight thermal arrest is observed. This thermal arrest is in fact so feeble that the repetition of the experiment does not show it again, due, probably, to the decreased sensitivity of the observations, through the formation of scale inside the sample. However, if the sample is freed from scale, similar thermal disturbance is again in evidence. Of much interest and significance is the observation that when this sample was allowed to cool at a rate of approximately 8 degrees Cent., per minute, no corresponding disturbance was observed. It is not possible, at least at the present time, to assign to the thermal disturbance on heating a definite explanation: it might be mentioned, merely as an observation, that when the alloys of this type, previously rendered thoroughly free from carbides, are reheated to approximately 850-900 degrees Cent. and allowed to cool either slowly or fast, such alloys will show minute carbides which have come out of solution and formed intricate formations.

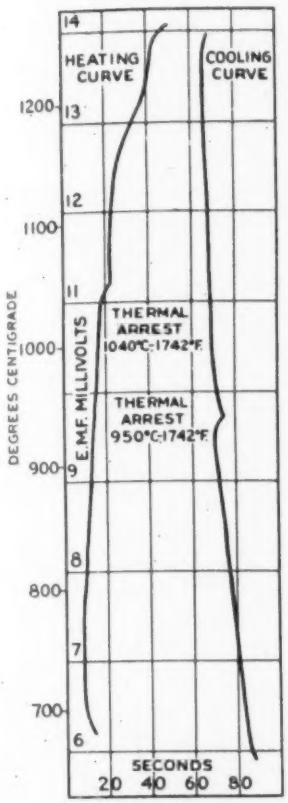


Fig. 3

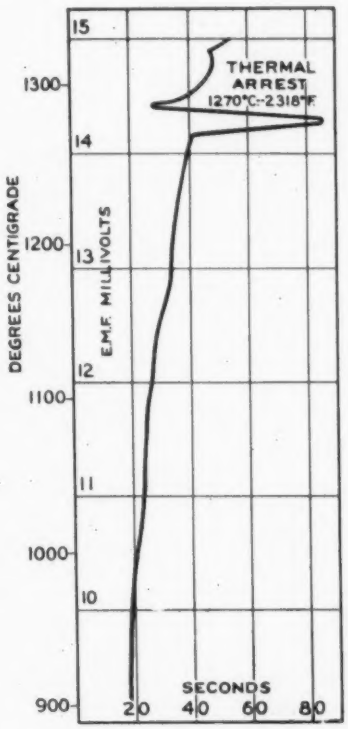


Fig. 4

often of troostite-like appearance, but sometimes in the conglomerations resembling well-known Widmanstatten patterns. One might, naturally, be tempted to the assumption that the coincidence between carbide re-precipitation at about 850 degrees Cent. and a thermal anomaly (also at about 850 degrees Cent.) is too pointed to be ignored. The absence of the thermal disturbance on cooling does not add much credence to this assumption.

From 850 degrees Cent. up no thermal arrest occurs until the temperature of 1155-1175 degrees Cent. (2110-2150 degrees Fahr.) is reached. At this temperature a pronounced but quite irregular disturbance sets in, invariably found on the repetition of the experiment. This disturbance takes place very close to that temperature at which the relative amount of gamma iron, as was shown by metallographic studies, is very markedly increased, delta iron being in decidedly subordinate position.

The thermal analysis was not carried high enough to determine the position of the "solidus" line.

The cooling curve taken from the same alloy, does not show thermal disturbances or arrests of any significance from 1300 degrees Cent. (2370 degrees Fahr.) down to room temperature.

Another alloy, No. 469, belonging to the same series of chromium content, but containing 0.62 per cent carbon has also been investigated with the results

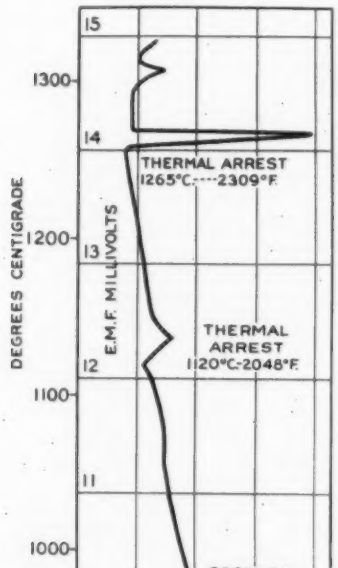


Fig. 5

shown in Fig. 2. The readings, as obtained, are plotted beginning at about 600 degrees Cent. and all way up to about 1350 degrees Cent. (2460 degrees Fahr.) at which temperature the point of eutectic formation is passed. As was the case with the previous alloy a slight disturbance occurs at about 850 degrees Cent. the rest of the curve below and above (up to 1350 degrees Cent.) this temperature being perfectly regular.

The position of the thermal arrest at 1350 degrees Cent. (2460 degrees Fahr.) coincides quite well with the dividing line between pure gamma iron area and the area in which eutectic is present. (See Fig. 51.) The position of this line (at least for the alloy in question) is placed quite correctly, although the accurate determination of it was made after the diagram was constructed.

It might have been expected that the disturbance at 1350 degrees Cent. should be of greater magnitude.

The cooling curve for the same alloy shows a definite point corresponding to one at 1350 degrees Cent. on heating, but from that temperature and all the way down to room temperature no further thermal arrest has been noticed, similarly to the alloy No. 468.

In Fig. 3 are reproduced a heating and a cooling curve for the alloy No. 2835, containing 27.90 per cent chromium and 0.63 per cent carbon. On heating a slight thermal disturbance is observed beginning at 1040 degrees Cent. (1905 degrees Fahr.). This point is quite close (on the temperature scale) to the lower horizontal line of the diagram in Fig. 52, and might be taken as representing the formation of the first sufficient amounts of gamma iron. There is, however, a discrepancy of about 200 degrees Fahr. in magnitude and this discrepancy is not overlooked.

A point, taken to correspond to the 1040 degrees Cent. one on heating, is

found at 950 degrees Cent. (1740 degrees Fahr.) on cooling. No other thermal arrests were observed either above or below the two points just mentioned. An attempt to determine "solidus" line did not yield sufficiently accurate and reliable results to incorporate them in the critical curve. It did show, however, that the position of "solidus" line as indicated in Fig. 52 is not far from its true position.

While investigating the alloy No. 3535 and others of the same chromium content, namely 33-35 per cent, no very evident thermal arrest (on heating) was detected, although certain irregularities in the potentiometer readings were indeed observed (Fig. 4) when the temperature of the sample was about 1200 degrees Cent. (2190 degrees Fahr.). Had it been possible to establish a sharply defined critical point at that temperature, it would correspond very well, indeed, to the lower horizontal line in Fig. 53, which depicts the appearance of first gamma iron grains.

The substantiation of the position of this line is found in the well-marked critical point, occurring on cooling, the peak of the point correspondng to 1120 degrees Cent. (2050 degrees Fahr.). The relatively lower position of this point (as compared with the position of the line at 2200 degrees Fahr. (See diagram Fig. 53) is, of course, readily explained by hysteresis.

Both heating and cooling curves for this alloy gave a very accurate datum in regard to the temperature at which a eutectic ("solidus" line) is formed. This temperature is found to be 1270 degrees Cent. (2320 degrees Fahr.) on heating and 1265 degrees Cent. (2310 degrees Fahr.) on cooling. In consequence, the position of the eutectic formation, as is shown in Fig. 53, should be somewhat lowered.

DISCUSSION

Written Discussion: By W. P. Sykes, metallurgical engineer, Cleveland Wire Works of General Electric Company, Cleveland.

As a study of a ternary alloy system, this paper offers several features worthy of commendation. The composition range involved is not too wide for thorough investigation. The methods employed are subject to satisfactory control, and the manner of presentation draws a well-defined picture of the constitutional relations existing in the regions under investigation. It may be well accepted, I believe, as a pattern by future workers in similar fields.

To the best of my knowledge there is here presented for the first time visual evidence of the formation of gamma iron from the solid delta and carbon-rich liquid phases upon cooling a steel, liquefied wholly or in part.

This type of reaction, known as a peritectic reaction, I have many times observed in tungsten-rich and molybdenum compositions in certain binary alloy systems containing these metals.

The structure shown in Fig. 17 of this paper is an especially good example of the result of such a reaction. In this case the gamma-rich phase is forming as a crust about the centers of the solid delta phase.

It would be interesting to learn what changes might occur in the troostitic areas were the alloys to be cooled less rapidly than by the drastic quench. What might develop in such areas if, after heating to 2400 degrees Fahr. (as

the specimen shown in Fig. 20) the specimen were to be cooled to say 2000 degrees Fahr. and held there for some time before quenching.

Would these erstwhile "troostitic" areas in the gamma regions be marked by carbide particles of visible size after some such treatment?

Written Discussion: By Dr. W. H. Hatfield, director, Brown-Firth Research Laboratories, Sheffield, England.

I have read this paper with very great interest, and should like to congratulate the authors upon the excellence of the metallographic work, particularly Figs. 14, 16 and 19, which do indeed represent a very high standard of technique. The authors also make excellent use of the different information obtained with the various etching media. I think it can be agreed that the authors make out a good case for their proposed diagrams, although some of the lines, they will contend, have been introduced with comparatively little evidence to support their actual position and shape. For instance, in Fig. 53, the section of the ternary diagram for 33 per cent chromium, the line separating the wholly solid from the partially liquid zone has been drawn at about 2410 degrees Fahr., although the two points just below this line apparently indicate the presence of some liquid phase at temperatures below this. Further, the trend of the position of this line with increase in chromium, as judged from the 22 and 28 per cent chromium diagrams, indicates that the line should really be at something less than 2400 degrees Fahr. Such points are, however, of minor order, and I should like to thank the authors for their extremely valuable contribution to the constitution of the iron-chromium-carbon alloys.

Written Discussion: By Dr. O. W. Ellis, director of metallurgical research, Ontario Research Foundation, Toronto, Canada.

One of the most difficult things for human beings is to break away from forms of expression which have become not merely antiquated, but distinctly confusing.

In certain circles, which need not be specified, "what is good enough for Father is good enough for me," and apparently in metallurgical circles as well "old custom" makes this life more sweet. In the writer's opinion, this adherence to what should be obsolete in metallurgical phraseology is due, fortunately, not to any "dyed-in-the-wool" belief that the terms employed have authoritative sanction, but rather to fears of confusing issues by changes in terminology.

The time has now been reached, however, when such fears should be abandoned and some radical action be taken if this very confusion is to be avoided. How, otherwise, is the unfortunate student to understand what is being placed before him in the literature? He, after all, is the one who should first be considered by those presenting material to the scientific societies, most of which must not only be read, but must be understood and absorbed by the undergraduates and graduates of the schools and universities throughout the world if they are to play a worthy part in the world's work.

The speaker has reason to believe that pure iron in the solid state can exist in two crystallographic forms—in one, the atoms being arranged on a body-centered cubic space lattice, in the other, on a face-centered cubic space lattice. So far as can be ascertained, what, for convenience, may be referred

to as body-centered iron, or B. C. iron exists at temperatures below about 905 degrees Cent. (the A_3 point) and above about 1410 degrees Cent. (the A_4 point). That the B. C. iron which exists below the A_3 point is essentially similar to, if not identical with, that which exists above the A_4 point seems now to be generally conceded—the fact that certain elements (notably aluminum, chromium, molybdenum, silicon and tungsten) bring about first the approach, then the coalescence, and finally the extinction of these points, being used as evidence in support of this view.

At the present time, B. C. iron below the A_3 point is generally referred to as alpha iron, while that above the A_4 point is referred to as delta iron. The use of the term "beta iron" for paramagnetic B. C. iron appears to have been almost universally discontinued. For purposes of this discussion, therefore, the term will be assumed to have passed into the limbo of lost expressions.

In view of the identity of the alpha and delta phases it seems unfortunate that in the normal growth of our knowledge of iron it should have been necessary to introduce distinguishing names for these phases. It seems more logical, in the light of our present knowledge, to refer to both alpha iron and delta iron as B. C. iron the natural result of this action being, of course, the substitution of the term "F. C. iron" for gamma iron, F. C. iron meaning iron possessed of a face-centered cubic lattice arrangement of atoms. F. C. iron (née gamma iron) exists at temperatures between the A_3 and A_4 points.

In the discussion of any of the *alloys* of iron, the use of such terms as alpha iron, gamma iron, and delta iron to describe microconstituents is to be strongly deprecated. In describing the constituents present in this series of ternary alloys of iron and carbon, for example, even Krivobok and Grossmann have erred though they may well be freed from blame in view of the confusion of tongues which characterizes the present age of ferrous metallography. Witness the fact that in certain of the beautiful photomicrographs which grace this study (Figs. 13, 14, 15, 16, and 19 in particular may be noted), these authors refer to alpha iron, delta iron, and gamma iron as coexisting at room temperature. This, if the writer mistakes not, is a physical impossibility, for alpha iron, and delta iron, as B. C. iron (as he would now suggest they should both be called), and gamma iron, or F. C. iron (as he would now suggest it should be called), can coexist at atmospheric pressure at either of two temperatures only—those temperatures corresponding to the A_3 and A_4 points. The coexistence of these two phases in any *alloy* is out of the question, though the coexistence of solid solutions of certain elements in both the B. C. and the F. C. phases of iron is, of course, possible. The delta iron and gamma iron of Krivobok and Grossmann's beautiful photomicrographs are, of course, solid solutions of carbon and chromium in B. C. and F. C. iron respectively. That this is their view of the nature of these constituents is made clear in their paper, though it is not specifically mentioned. The expert can infer that such is their opinion; the confusion of the tyro, however, may well be worse confounded by the variety of terms used to denote one and the same constituent.

Further consideration of these photomicrographs raises another interesting point. It has been the speaker's custom for many years to refer to the solid solution of carbon in B. C. iron as ferrite. Some have restricted this term—

ferrite—to B. C. iron (alpha iron), but the speaker has, in his lectures on physical metallurgy during the last five years at least, consistently avoided confusing B. C. iron with the solid solutions of carbon in B. C. iron. The former he has referred to as "alpha iron" (he would now prefer to call it B. C. iron), the latter he has referred to as "ferrite." To ternary alloys containing iron and carbon, and having a body-centered cubic lattice structure, he has applied such terms as "chromium ferrite," "silicon ferrite," etc., according to the third element present in the material. The principle can, of course, be extended to cover the use of more complex alloys as well.

By using the term "ferrite" to denote the solid solution of carbon in B. C. iron, and the solid solution *only*, much confusion can be avoided. By referring to alpha iron, beta iron, and delta iron as B. C. iron, still further trouble can be prevented.

Thus far, the use of the term "austenite" has been generally, though not universally, applied to the solid solution of carbon in B. C. iron. The speaker would suggest that the term "austenite" be applied to the binary solid solution only, and that for the solid solutions of carbon and any one or more elements in F. C. iron the terms "M-austenite," "MM_1-austenite," etc., be employed, where M, M_1, etc., refer to the special elements in the steel. For example, the solid solution of 22 per cent chromium, 0.5 per cent carbon and 77.5 per cent F. C. iron existing at 1200 degrees Cent. would be referred to as chromium-austenite.

The coexistence of chromium-ferrite and chromium-austenite is clearly possible. It seems that the use of such terms to describe the constituents in question is far to be preferred to that of the terms "delta iron" and "gamma iron" which have been used in this connection by the authors.

Insofar as the iron-carbon system is concerned, it may be noted that the coexistence of ferrite and austenite is possible within two fields of equilibrium, because, on the one hand, B. C. iron can exist at both high (above A_4) and low (below A_3) temperatures, and, on the other hand, the transformation of B. C. iron to F. C. iron (and vice versa) at A_3 is lowered, and that of F. C. iron to B. C. iron (and vice versa) at A_4 is raised as the result of mixing carbon with the pure metal.

In conclusion, the writer would extend his apologies to Dr. Krivobok and Mr. Grossmann for the liberty he has taken of using their paper as a winnowing-fan. Their paper, being, as it is, full of valuable information, and presenting, as it does, work of the very highest character, does not deserve to be marred by the use of terms savouring of inexactitude. Nor, for that matter, do any other papers merit this fate. If, therefore, this well-meant criticism may lead to the use of a more precise phraseology in metallographic circles, not only will the speaker be happy, but the authors, whom he knows will forgive him for using them as "goats," will be well repaid.

Written Discussion: By O. V. Greene, metallurgist, Carpenter Steel Company, Reading, Pa.

This paper represents an amazing amount of painstaking work, and I feel that little can be added to the data presented. However, for the benefit of future investigators of alloys of this nature, would it not be advisable to stress

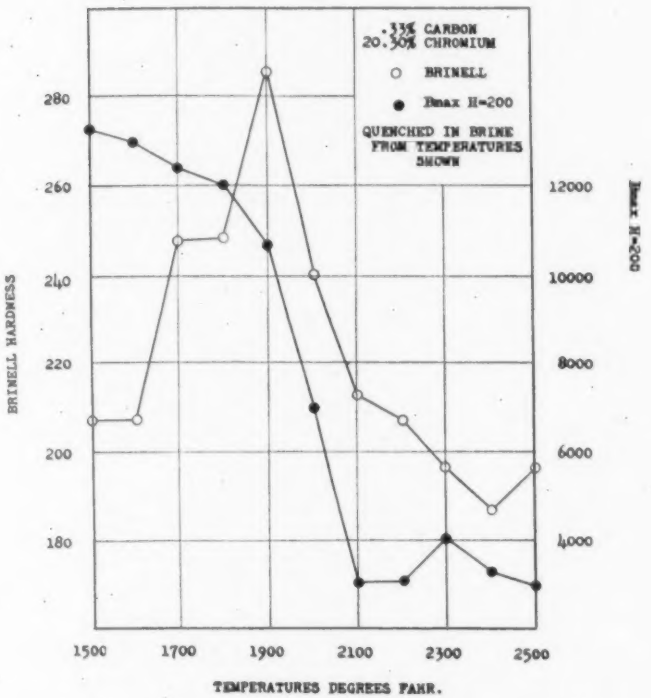


Fig. 1—Hardness and Magnetic Properties After Treatment B.

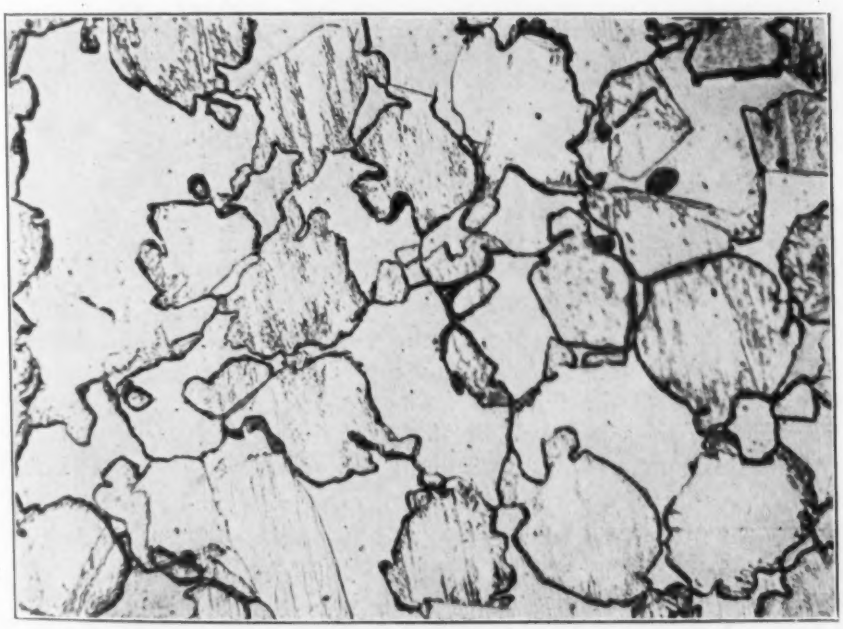


Fig. 2—Structure Obtained after Treatment B, Quenched from 2400 degrees Fahr. Etched with Aqua Regia. Constituents: Chromium ferrite, Chromium austenite and Eutectic. × 250.

Table I
Constituents Obtained with Different Rates of Heating and Lengths of Soaking

TREATMENT A Specimens placed in hot furnace, held 20 min. at temperature	TEMPERATURES DEG. FAHR. Brine quenched	TREATMENT B Specimens heated with furnace, held one hour at temperature
Chromium-ferrite and carbides	Annealed	Chromium-ferrite and carbides
Chromium-ferrite and carbides	1500	Chromium-ferrite and carbides
Chromium-ferrite and carbides	1600	Chromium-ferrite and carbides
Chromium-ferrite and carbides	1700	Chromium-ferrite, trace of chromium - austenite, carbides diffusing
Chromium-ferrite and carbides	1800	Chromium-ferrite, chromium-austenite and carbides
Chromium-ferrite, trace of chromium - austenite and carbides diffusing	1900	Chromium-ferrite, chromium-austenite and carbides
Chromium-ferrite, chromium-austenite and carbides	2000	Chromium-ferrite, chromium-austenite and carbides
Chromium-ferrite, chromium-austenite and carbides	2100	Chromium - austenite, some chromium-ferrite carbides entirely diffused
Chromium - austenite, some chromium-ferrite, carbides diffused	2200	Chromium - austenite, some chromium-ferrite, no carbides
Chromium - austenite, some chromium-ferrite, no carbides	2300	Chromium - austenite, some chromium-ferrite, no carbides
Chromium - austenite, some chromium - ferrite, no carbides	2400	Chromium - austenite, some chromium-ferrite, eutectic, no carbides
Chromium - austenite, some chromium-ferrite, eutectic, no carbides	2500	Chromium - austenite, some chromium-ferrite, eutectic, no carbides

the importance of taking due consideration of the extreme sluggishness of such alloys.

The results of our experience on an alloy containing 0.33 per cent carbon, 20.30 per cent chromium and 0.96 per cent copper may be taken as an example of the effect of too rapid heating and too short soaking. The microstructures obtained by rapid heating and insufficient soaking as compared to those obtained by slow heating and long soaking are given in Table I of this discussion. It may be noted that specimens with treatment A do not show the same structural changes as with treatment B until 100 or 200 degrees Fahr. higher have been reached. Is it therefore unreasonable to suppose that with extremely slow heating and sufficient soaking that the apparent critical point at 1800 degrees Fahr. can be further lowered?

The microstructures given in Table I as the result of treatment B are seen to be in close agreement with the authors' results. Fig. 1 is given as further confirmation of the hardness and magnetic properties of this alloy. Fig. 2 of this discussion shows the existence of eutectic as reported in the paper.

Author's Reply

We wish to thank Mr. Sykes for his courteous comment, especially in view of his own extensive work in similar fields.

We are very much interested in the remark on visual evidence of the formation of gamma iron from the solid delta iron and carbon-rich liquid phase upon cooling a steel. This particular branch of work was followed a little further, as is kindly suggested by Mr. Sykes, and the only difference which we noted between the samples drastically quenched from 2400 degrees Fahr. and those slowly cooled to 2000 degrees Fahr. and held there for eight hours was a relative increase in the size of carbide particles, though eight hours is of course not a long time. We are, indeed, obliged to Mr. Sykes for his kind remarks concerning the paper.

We deeply appreciate the remarks sent to us by Dr. Hatfield. We noticed, ourselves, a very deplorable mistake in the diagram shown on page 35, as pointed out by him. We noticed that we drew a line where it does not belong, but it was too late to correct it. It is obvious that this line should be below that phase which is indicated by "LL," the latter standing for liquefied metal. We regret this unfortunate omission on our part and wish to thank Dr. Hatfield for reading our contribution with as much attention as he apparently has.

The point raised by O. V. Greene is worthy of attention. Indeed, Mr. Greene is obviously quite right when he states that chromium alloys, and, as a matter of fact, many others including chromium-nickel, chromium-nickel-silicon, chromium-nickel-aluminum, and others, are extremely sluggish, and the problem of studying the constituents of such steels at relatively low temperatures, let us say around 1600 to 1800 degrees Fahr., becomes one of serious difficulties. We think that one way to obtain the most reliable results is to subject sluggish steels to a high temperature treatment (avoiding, however, the formation of liquefied phase) and then subject such samples to prolonged treatments at lower temperatures without intermediate cooling. In the present work, the heating time at low temperatures was sometimes as long as eight hours. Moreover, we had a number of samples first heated to a high temperature, then slowly cooled with the furnace to a temperature of much lower magnitude and rapidly quenched from the latter. The results on the whole were quite in accordance with the observations, as noted in the paper. We are indeed glad that our results are in close agreement with those obtained by Mr. Greene.

The remarks by our esteemed friend, Dr. O. W. Ellis, present us with the opportunity of accepting his well-taken criticism in a worthy cause. We cannot but agree with Dr. Ellis that the confusion in the phraseology as applied to the scientific study of metals has reached such proportions that it really must be corrected. In fact, one of the authors has made—only a short time ago—a plea on the very same subject.* However, perhaps the question of phraseology will be settled by, if we may suggest, some sort of committee or another equally qualified body, and in the meantime we have admittedly been guilty of some looseness in phraseology. Although we seriously considered the matter of definitions before we put our results in print, we failed, as Dr. Ellis rightly observes, to make it clear enough. We thank Dr. Ellis most sincerely for his kind, clever, friendly, and important criticism.

*V. N. Krivobok, "A Study of the Constitution of High Manganese Steels," TRANSACTIONS, American Society for Steel Treating, June, 1929, Vol. XV, No. 6, p. 918.

INFLUENCE OF NICKEL ON THE CHROMIUM-IRON-CARBON CONSTITUTIONAL DIAGRAM

By V. N. Krivobok and M. A. Grossmann

Abstract

The microstructures of chromium-nickel-iron alloys are studied, examining the effect of successive additions of nickel to 18 per cent chromium irons with various carbon contents. The alloys can be called 18-2, 18-4, 18-8 and 18-12, containing in all cases 18 per cent chromium, the nickel contents being respectively 2, 4, 8 and 12 per cent. In each group several carbon contents were included, ranging between 0.05 and 0.60 per cent.

The extent to which nickel widened the austenite field is illustrated in analogues of iron-carbon diagrams, the "iron" in these cases being alloyed with 18 per cent chromium and the added amount of nickel. Nickel widens the range of temperature in which austenite is stable. It also widens the range of carbon content in which austenite alone is the stable phase, and narrows the range of carbon content in which the stable structure is an association of ferrite and austenite.

Some magnetic data and certain hardness values are included, together with a discussion of the precipitation of carbides upon heating at 1200 degrees Fahr.

THE present investigation has to do with the constitutional diagrams of certain high chromium-nickel steels, that is to say, a series of "iron-carbon diagrams" representing the structures found in steels containing certain large amounts of chromium and nickel.

The amounts of alloy chosen for study call for some explanation, as does also the manner of presentation of the data.

The amounts of alloy are in the ranges of composition which are at the moment commanding attention in stainless irons. Most of the stainless materials in use today will be found in two groups of composition, the plain chromium group, containing variously

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. Of the authors, who are members of the society, Dr. V. N. Krivobok is associate professor of metallurgy at Carnegie Institute of Technology at Pittsburgh, and Dr. M. A. Grossmann is metallurgical engineer, Republic Steel Corp., Canton, Ohio. Manuscript received June 23, 1930.

from 13 to 18 per cent chromium, and the so-called 18-8 group, containing about 18 per cent chromium and about 8 per cent nickel. It is these ranges of composition that are studied.

Now, the structures themselves which are found in these steels are quite the same as those found in plain carbon steels (ferrite, austenite, etc.). Furthermore, again as in plain carbon steels, the proportions of those structures and the temperatures at which they are stable are affected by the carbon content of the steel.

It is not surprising to find, therefore, that a useful correlation of the structures found could be made in the form of constitutional diagrams similar to the well-known iron-carbon diagram. The construction of these constitutional diagrams was carried out as described below.

As regards the chromium steels without nickel, a study of a series of such steels had already resulted[1] in the construction of the diagrams of Fig. 1. These show the influence of chromium in modifying the positions of the structural fields in the iron-carbon diagram. The diagrams of Fig. 1 are sections of the ternary iron-chromium-carbon diagram, these sections being planes parallel to the iron-carbon equilibrium diagram. Although they are not *equilibrium* diagrams, it is useful to consider them as being iron-carbon diagrams in which pure iron is replaced by alloyed iron containing the indicated percentage of chromium. Examined as a group, they show how an increase in the chromium content causes the region of [delta ferrite plus austenite] to approach in temperature the region of [alpha ferrite plus austenite] and finally to merge with it; they show how further addition of chromium contracts the temperature range in which the mixture of ferrite and austenite is stable, while at the same time widening the range of carbon content in which it is found; and finally they show how addition of chromium narrows the range both of temperature and of carbon content in which austenite is the only stable phase.

The construction of the above diagram is based on microscopic examination of the structures, the structures having been preserved from the high temperatures by quenching. The examination of the above chromium alloys, as well as that of the chromium-nickel alloys about to be described, was carried out as follows. The experimental ingots, of about 20 pounds each, were forged to 7/8 inch square, and

[1] Krivobok and Grossmann, "Constitutional Diagram of Chromium-Iron-Carbon Alloys", TRANSACTIONS of the American Society for Steel Treating, Vol. XVIII, July 1930, p. 1.

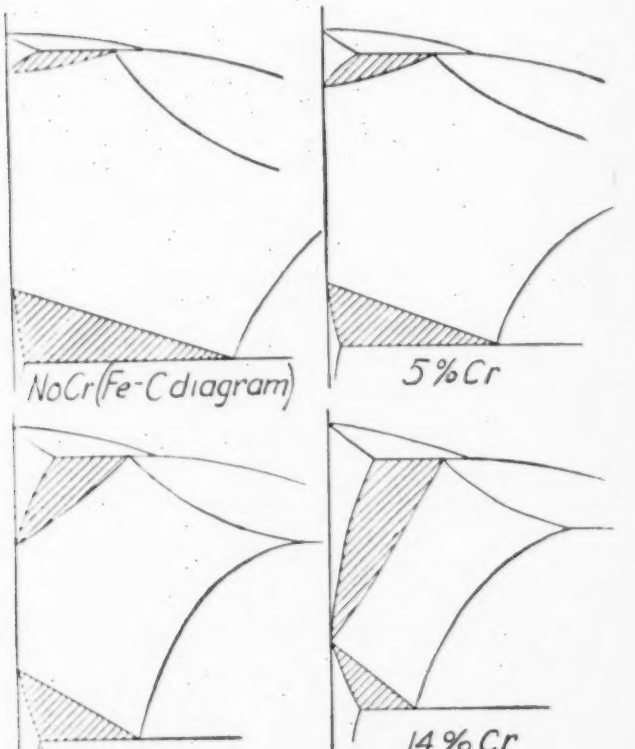


Fig. 1—Curves Showing the Effect of the Presence of Chromium on the Iron-Carbon Constitutional Diagram.

heated for 7 to 8 hours at about 1800 degrees Fahr., and furnace cooled. Slices of these bars were heated to the respective temperatures, held at temperature for one hour (or occasionally longer) and then quenched in water or iced brine. The slices were then cut in half and examined under the microscope. These same pieces were also tested for hardness, and the averages of the hardness values for the alloys containing 2, 4 and 8 per cent nickel are given in Figs. 1A to 1C. The structures which were observed under the microscope represented quite accurately, it is believed, the phases present at the moment of quenching. It had been found that one hour was sufficient time to bring about equilibrium above 1600 degrees Fahr. (875 degrees Cent.), and it is known that these alloys are so sluggish that a quench preserves quite undisturbed the structures present at high temperatures.

In constructing the diagram for each particular group of alloys, the various carbon contents were plotted as abscissae, and on the temperature ordinates were recorded the structures found at the respective temperatures. It was simple, then, to draw in the lines bounding the respective fields in which various structures were stable. The results in the case of chromium are found in the pseudo iron carbon diagrams of Fig. 1, which shows the influence of chromium in modifying the structures found in iron-carbon alloys.

Now it is obvious that the same procedure can be applied when other alloying elements are present, so long as the behavior of the alloying element is analogous to that of the iron, so that such alloy as is present can be considered to replace part of the iron. In the case of the data about to be presented, chromium is present in constant amount (18 per cent), with nickel in various amounts from zero to 12 per cent. Analogues of iron-carbon diagrams will again be constructed, the pure iron of the iron-carbon diagram being replaced by

1. iron alloyed with 18 per cent chromium
2. iron alloyed with 18 per cent chromium and 2 per cent nickel
3. iron alloyed with 18 per cent chromium and 4 per cent nickel
4. iron alloyed with 18 per cent chromium and 8 per cent nickel
5. iron alloyed with 18 per cent chromium and 12 per cent nickel

In the examination of each group, a number of alloys of different carbon content are studied, the alloy content for that group being held approximately constant, with variations in carbon from 0.05 to about 0.60 per cent.

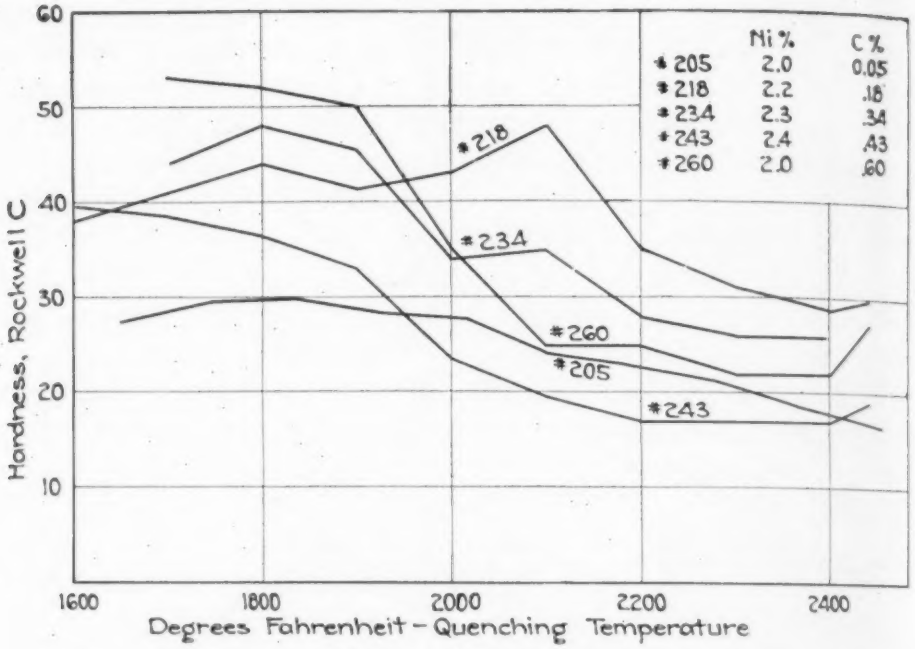


Fig. 1A—Rockwell C Hardness versus Quenching Temperature Curves for Alloys with Varying Nickel Contents.

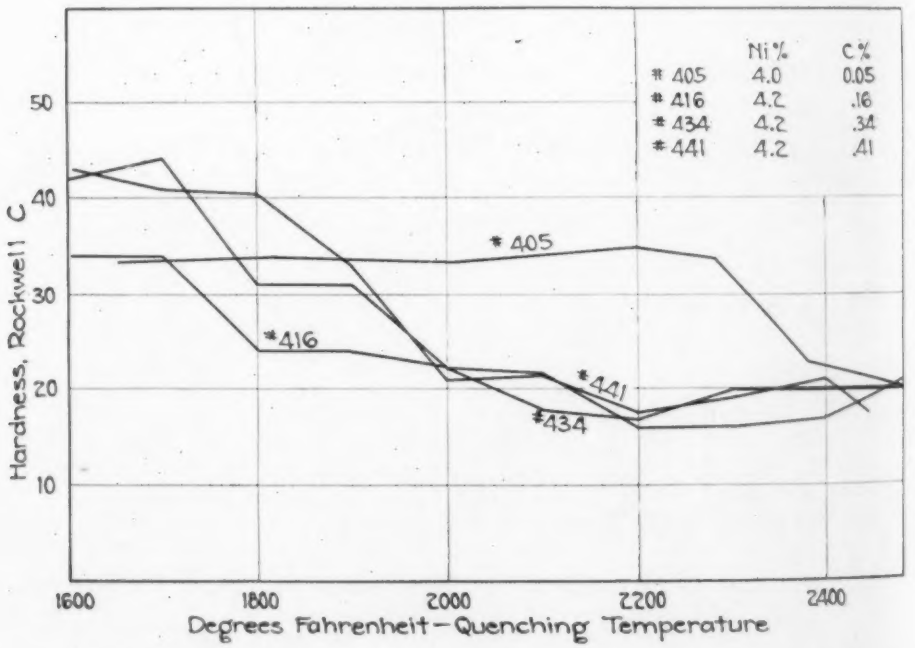


Fig. 1B—Rockwell C Hardness versus Quenching Temperature Curves for Alloys with Varying Nickel Contents.

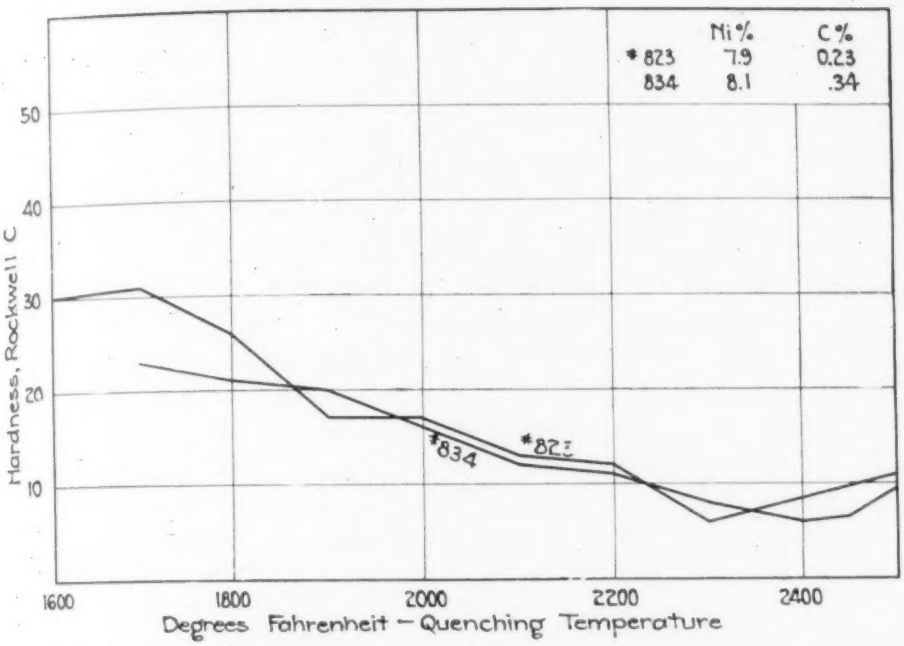


Fig. 1C—Rockwell C Hardness versus Quenching Temperature Curves for Alloys with Varying Nickel Contents.

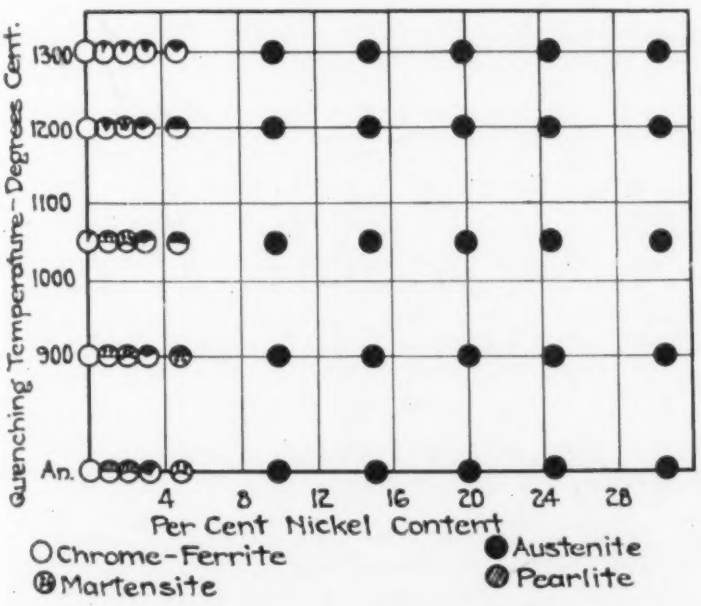


Fig. 2—Structures Reported by Bain and Griffiths, Various Alloy Contents, Chromium Approximately 20 Per Cent, Carbon About 0.15 Per Cent.

So far as previous work is concerned, the authors are aware of

only one systematic survey, that of Bain and Griffiths.[2] That work covers a vastly wider range of alloy contents than does the present paper, namely, from 5 to 60 per cent chromium, and from zero to 30 per cent nickel, the carbon content being in the neighborhood of 0.15 per cent. The present paper undertakes on the other hand to study the effect of carbon, but in a much more restricted range of alloy contents. Fig. 2 is reproduced from the paper of Bain and Griffiths, and summarizes their results in that range of alloys which corresponds most closely to the alloys studied in the present paper.

The results of the present work are grouped in Tables I, II, III and IV. Those tables list the structures which were found in the various steels when they had been heated to the temperatures indicated, had been held there at least one hour, had been quenched and had then been cut and examined under the microscope.

The structures which are referred to in the Tables are the following:

Alpha ferrite: chromium and nickel-bearing alpha iron containing some carbon in solution.

Delta ferrite: chromium and nickel-bearing delta iron containing some carbon in solution (for convenience in designation, the authors subscribe to the custom of distinguishing by the name "delta" the high-temperature form of body-centered iron, often formed by heating beyond a temperature range where gamma iron or austenite is stable, into the range where delta iron is stable).

Austenite (symbol A): chromium and nickel-bearing gamma iron with carbon in solution.

Carbides (symbol C): the complex alloy carbides corresponding in behavior but not in composition to iron carbide Fe_3C. They are believed to contain from 30 to 60 per cent chromium and from 5 to 20 per cent nickel.

Liquefied metal (symbol L): some of the materials, especially those highest in carbon, when treated in the higher ranges of temperature, became partially molten. The regions which became molten always exhibited a characteristic structure and are thus specially designated.

The microstructures to which reference is made are illustrated in the accompanying photomicrographs. Since in many cases the structures bore marked resemblance to one another, it has been considered sufficient to show typical cases. Where individual instances are chosen for illustration, they are mentioned specifically in the tables.

The data given in Tables I, II, III and IV are given more concisely and correlated in Figs. 3, 4, 5 and 6. These are the analogues

[2]Bain and Griffiths "An Introduction to Iron-Nickel-Chromium Alloys", *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol LXXV, 1927, p. 166.

Table I

Base: 18 Per Cent Chromium-Iron Alloy

Nickel 2 Per Cent

Degrees Fahr.	Alloy No. 205 Carbon 0.05 Nickel 2.0 Chromium 17.4	Alloy No. 218 Carbon 0.18 Nickel 2.2 Chromium 17.1	Alloy No. 234 Carbon 0.34 Nickel 2.3 Chromium 16.7	Alloy No. 243 Carbon 0.43 Nickel 2.5 Chromium 17.5	Alloy No. 260 Carbon 0.60 Nickel 2.0 Chromium 18.2
2500 or 2450	Delta ferrite, plus austenite precipitated at grain boundaries and within grains. Magnetic. Fig. 8.	Traces of liquefied metal. Equal number of austenite and delta ferrite, austenite being decomposed into martensite. Magnetic.	Large austenitic grains with martensitic formations at grain boundaries and within grains. Also liquefied metal. Very slightly magnetic. Fig. 10.	Large size austenitic grains with numerous twins. Liquefied metal. Nonmagnetic.	Chrome austenite plus liquefied metal. Nonmagnetic. Fig. 11.
2400	As above. Magnetic.	No liquefied metal. Less delta ferrite. The structure consists of equiaxed austenite grains transformed into martensite. Magnetic.	As above. No liquefied metal. Very slightly magnetic.	As above. Nonmagnetic.	As above Nonmagnetic
2300	Delta grains plus grains of martensitic structure. Magnetic. Fig. 9.	As above. Smaller grain size. Magnetic.	Smaller austenitic grains, more martensite like constituents. Very slightly magnetic.	Austenitic grains, no liquefied metal.	Chrome austenite, martensite like constituent at grain boundaries. Slightly magnetic.
2200	As above. Magnetic.	Smaller grain size. Small rectangular areas (delta ferrite?) easily seen after light etch. Magnetic.	As above, smaller grain. Very slightly magnetic.	As above. Nonmagnetic.	As above. Slightly magnetic.
2100	As above. Magnetic.	As above. Magnetic.	As above, still smaller grain size. Nonmagnetic.	Much smaller austenitic grains. A few carbides. Nonmagnetic.	As above. More martensite. Slightly magnetic. Carbides Fig. 12.
2000	As above. Magnetic.	All martensite. Magnetic.	Practically the same as above. Structure difficult to reveal carbides. Nonmagnetic.	As above. More carbides. Nonmagnetic.	Martensite, some austenite and carbides. Fairly large grain size. Magnetic.
1900	As above. Martensite predominates. Magnetic.	As above. Magnetic.	Grains no longer resemble austenite, but all martensite carbides. Magnetic.	Change in structure: very small grains with numerous carbides in streaks and around grain boundaries. Very slightly magnetic.	Martensite and carbides. Fine grain size. Magnetic.
1800	As above. Magnetic.	As above. Magnetic.	As above. Magnetic.	As above. Magnetic.	As above. Magnetic.
1700	As above. Magnetic.	As above. Magnetic.	As above. Magnetic.	As above. More magnetic.	As above. Magnetic.
1600		As above. Magnetic.		As above. Very magnetic.	

Table II

18 Per Cent Chromium-Iron Series
Nickel 4 Per Cent

Degrees Fahr.	Alloy No. 405 Carbon 0.05 Nickel 4.0 Chromium 17.4	Alloy No. 416 Carbon 0.16 Nickel 4.2 Chromium 17.7	Alloy No. 434 Carbon 0.34 Nickel 4.2 Chromium 17.5	Alloy No. 441 Carbon 0.41 Nickel 4.2 Chromium 17.4
2450 to 2500	Large grains of delta ferrite, and austenite precipitating on cooling.	Large austenitic grains probably some delta ferrite. Liquefied metal. Slightly magnetic.	Austenitic grains with twins. Numerous liquefied metal-like at grain boundaries.	Large amount of liquefied metal. Large austenitic grains. Fig. 15.
2400	As above.	No liquefied metal. Large austenitic grains. A constituent at grain boundaries and within grains that can be delta ferrite. Very slightly magnetic. Fig. 13.		Liquefied metal in small amounts at grain boundaries and within grains. Large austenitic grains.
2300	Delta ferrite plus martensitic grains. (Change in structure)	Large austenitic grains. No liquefied metal. Nonmagnetic.	Austenite only.	No liquefied metal. Austenite only.
2200	Less delta ferrite, more martensitic grains.	Large austenitic grains. Numerous twins. Nonmagnetic.	Austenite only.	As above.
2100	As above.	Much smaller austenitic grains. Nonmagnetic	Smaller austenitic grains: carbides seem to be present.	Smaller austenitic grains. Some individual carbides.
2000		Still smaller austenitic grains. Nonmagnetic	Austenite, more carbides.	Change in austenitic grain size: numerous carbides at grain boundaries and in streaks.
1900		Very fine austenitic grains, difficult to distinguish. Nonmagnetic.	Change in structure: small grains of austenite.	As above.
1800		Very fine structure, austenitic grains losing its identity. Nonmagnetic.	Same as above, in places looks like decomposition at grain boundaries.	Change in structure: massive carbides at grain boundaries, very small grain size.
1700	As above.	Grains difficult to bring out. Numerous carbides. Very slightly magnetic.	Same as above. Decomposition has taken place within grains so martensitic structure is obtained.	As above.
1600		Very numerous carbides. Well defined grains. Very magnetic. Fig. 14.		As above.

of iron-carbon diagrams, the iron being replaced by 18 per cent chromium iron together with (respectively) 2, 4, 8 and 12 per cent nickel.

The manner of constructing these diagrams is fairly obvious, when we consider the data given in the corresponding tables. A few words of explanation may, however, be in order, and we may comment briefly on Fig. 3 (2 per cent nickel). Alloy No. 205 (0.05

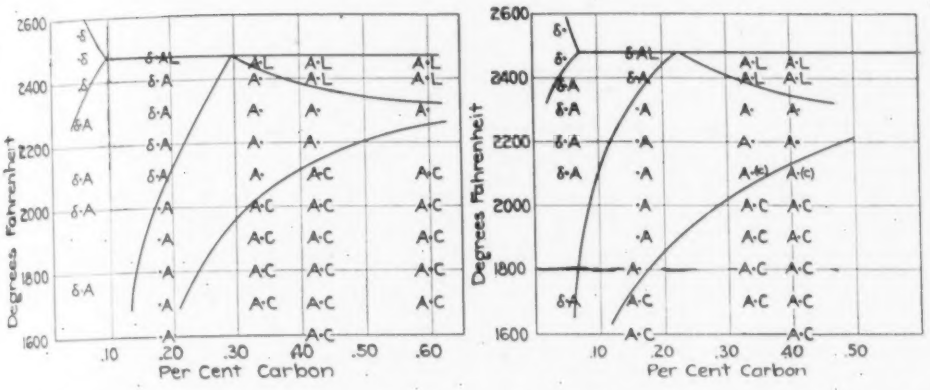


Fig. 3—Data of Table I Assembled into a Constitutional Diagram for Steels with 18 Per Cent Chromium plus 2 Per Cent Nickel.

Fig. 4—Data of Table II Assembled into a Constitutional Diagram for Steels with 18 Per Cent Chromium plus 4 Per Cent Nickel.

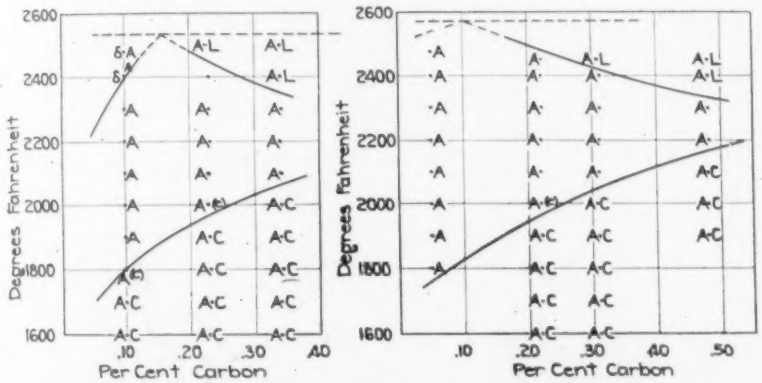


Fig. 5—Data of Table III Assembled into a Constitutional Diagram for Steels with 18 Per Cent Chromium plus 8 Per Cent Nickel.

Fig. 6—Data of Table IV Assembled into a Constitutional Diagram for Steels with 18 Per Cent Chromium plus 12 Per Cent Nickel.

per cent carbon) consists of delta ferrite only (the austenite which is visible having precipitated from the delta iron upon cooling), in the range above 2350 degrees Fahr. Below 2300 degrees Fahr. the structure is a mixture of delta ferrite and austenite. The line separating the field of pure delta from the field of mixed structures must therefore be drawn between 2300 and 2350 degrees Fahr. Its probable slope is judged from work on many other iron-carbon-alloy diagrams.

Alloy No. 218 (0.18 per cent carbon) shows a structure consisting of mixtures of delta and austenite from 2100 to 2400 degrees Fahr. Below 1900 degrees Fahr. the structure upon cooling is martensite (formerly wholly austenite) so that we again know that the line separating the field of mixed structures (delta plus ferrite)

Table III

18 Per Cent Chromium-Iron Alloy
Nickel 8 Per Cent

Degrees Fahr.	Alloy No. 810 Carbon 0.10 Nickel 8.0 Chromium 18.0	Alloy No. 823 Carbon 0.23 Nickel 8.0 Chromium 18.0	Alloy No. 834 Carbon 0.34 Nickel 8.1 Chromium 17.6
2450 to 2500	Large austenite grains with some delta ferrite.	Large austenite grains with liquefied metal. Murakami etches. Liquefied metal indicating presence carbides. Nonmagnetic. Fig. 17.	Large austenite grains with liquefied metal at grain boundaries and within grains. Nonmagnetic.
2400	Large austenite grains. Small amount of delta ferrite.		Large austenite grains, liquefied metal in one or two places only. Nonmagnetic.
2300	Austenite.	Typical austenite grains, numerous twins. Nonmagnetic.	Austenite grains, *no* liquefied metal. Nonmagnetic.
2200	Austenite.	Austenite, fairly large grain size. Nonmagnetic.	Change in grain size; all austenite. Nonmagnetic.
2100	Austenite. Fig. 16.	Austenite. Nonmagnetic.	As above. Nonmagnetic.
2000	Austenite.	Austenite, smaller grains, some carbides. Nonmagnetic.	Very marked change in grain size; carbides in streaks and all throughout. Nonmagnetic.
1900	Austenite.	Austenite, carbides around grain boundaries. Nonmagnetic.	Very fine grain size. Much increased carbides. Nonmagnetic.
1800	Austenite with faint precipitation of carbides.	As above. Nonmagnetic.	As above. Nonmagnetic.
1700	Austenite with some carbides.	As above. Nonmagnetic.	As above. Nonmagnetic.
1600	As above.	As above. Nonmagnetic.	Nonmagnetic.

from the field of pure austenite must pass in the neighborhood of 2000 to 2100 degrees Fahr. for this particular composition. Also, at 2450 to 2475 degrees Fahr. this alloy shows the first indication of melting, so that we know the peritectic line comes at about this temperature.

In alloy No. 235 (0.35 per cent carbon), we find only austenite from 2100 to 2400 degrees Fahr. At 2000 degrees Fahr. and below we find carbides associated with the austenite, and we know therefore that the hypereutectoid line must pass between 2000 and 2100 degrees Fahr. Also at about 2450 degrees Fahr. this alloy shows some liquefied metal, which is an index that the liquidus line lies between 2400 and 2450 degrees Fahr. The position of the hypereutectoid line and the position of the liquidus line is found for alloys No. 245 and No. 260 (0.45 and 0.60 per cent carbon) in the same manner as for alloy No. 235. The various lines in the diagram have thus been located in a quite simple manner. The same procedure is followed in the other diagrams.

It has been assumed by the authors that the diagram for 18 per

Table IV
18 Per Cent Chromium-Iron Series
Nickel 12 Per Cent

Degrees Fahr.	Alloy No. 1205 Carbon 0.05 Nickel 11.4 Chromium 17.8	Alloy No. 1222 Carbon 0.22 Nickel 12.0 Chromium 18.1	Alloy No. 1231 Carbon 0.31 Nickel 11.7 Chromium 18.1	Alloy No. 1248 Carbon 0.48 Nickel 11.3 Chromium 19.0
2500 or 2450	Large austenitic grains. No liquefied metal.	Large austenitic grains. No liquefied metal.	Large austenitic grains. Very little liquefied metal at grain boundaries.	Very large austenite. Plentiful liquefied metal. Fig. 20.
2400	Large austenitic grains.	Large austenitic grains.	Large austenite. No liquefied metal. See Fig. 18.	Very large austenite, pronounced liquefied metal.
2300	As above.	Small and large austenitic grains.	Austenite — large and small.	Fairly large austenite. No liquefied metal.
2200	As above.	Somewhat smaller grain size austenite.	Change in grain size: austenite.	Smaller austenite.
2100	As above. Much smaller grain size.	Very small austenitic grains.	Austenite, same as above.	Very marked change in austenitic grains, carbides.
2000	As above.	Very small austenitic grains with some carbides (?).	Very marked change in grain size: austenite plus carbides at grain boundaries.	As above.
1900	As above.	As above.	As above.	As above plus more carbides.
1800	As above. Forging outline seen.	As above.	Still finer austenitic grains: carbides at grain boundaries. Fig. 19.	
1700		As above. Some decomposition at grain boundaries.	Austenite difficult to reveal. Carbides.	
1600		As above.	Austenite and carbides throughout.	

cent chromium iron, without nickel, is substantially correct as given in Fig. 1. Adopting this view, we may now present the five "iron-carbon diagrams" shown in Fig. 7, for 18 per cent chromium iron with 0, 2, 4, 8 and 12 per cent nickel. If we compare the effect of nickel with the effect of chromium, we find that the nickel has an effect opposed to that of chromium. Quite contrary to the effect of chromium as outlined at the beginning of this paper (sixth paragraph), we find that nickel widens the temperature range in which mixtures of ferrite and austenite are stable, while at the same time narrowing the range of carbon content in which such mixtures are found; nickel also widens the range both of temperature and of carbon content in which austenite is the only stable phase.

The construction of the diagrams above 1600 degrees Fahr. has been seen to be a relatively simple matter. Although the alloys are very sluggish in their reactions, nevertheless, a heating procedure of an hour to an hour and a half suffices to establish an approximation

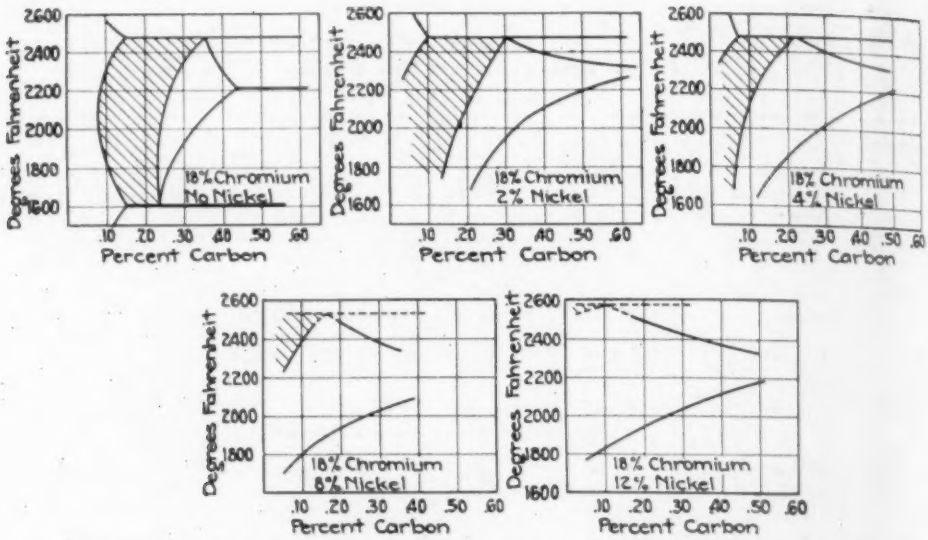


Fig. 7—Diagrams Illustrating the Influence of Nickel in Modifying 18 Per Cent Chromium Iron-Carbon Constitutional Diagram.

to equilibrium, when the heating temperature is above 1600 degrees Fahr.

Below this temperature of 1600 degrees Fahr., however, the diffusion becomes markedly slower as the temperature falls, which means that equilibrium is established only after extremely long periods of time, if at all. At temperatures of 800 to 1000 degrees Fahr. it will be seen that a heating time of 100 hours was insufficient for anything except a mere faint modification of the already existing structure.

A series of tests were made with the present alloys, covering magnetic data, microstructure and hardness. It will be seen that the data obtained are insufficient for the construction of any equilibrium diagram at lower temperatures, but certain observations are worthy of consideration.

Table V lists the hardness and a magnetic index for each treatment of the alloys from 800 to 1400 degrees Fahr. The treatments at 800 to 1400 degrees Fahr. were carried out on the alloys which were in the "as-forged" condition, that is to say, after the alloys had been cooled in air from the forging temperature in the neighborhood of 1900 degrees Fahr.

The hardness values are assembled in the diagrams of Fig. 21. In these diagrams a tendency to soften is probably accounted for largely by the softening of a martensitic structure, but also partly in some cases by the removal of forging strains and in other cases by

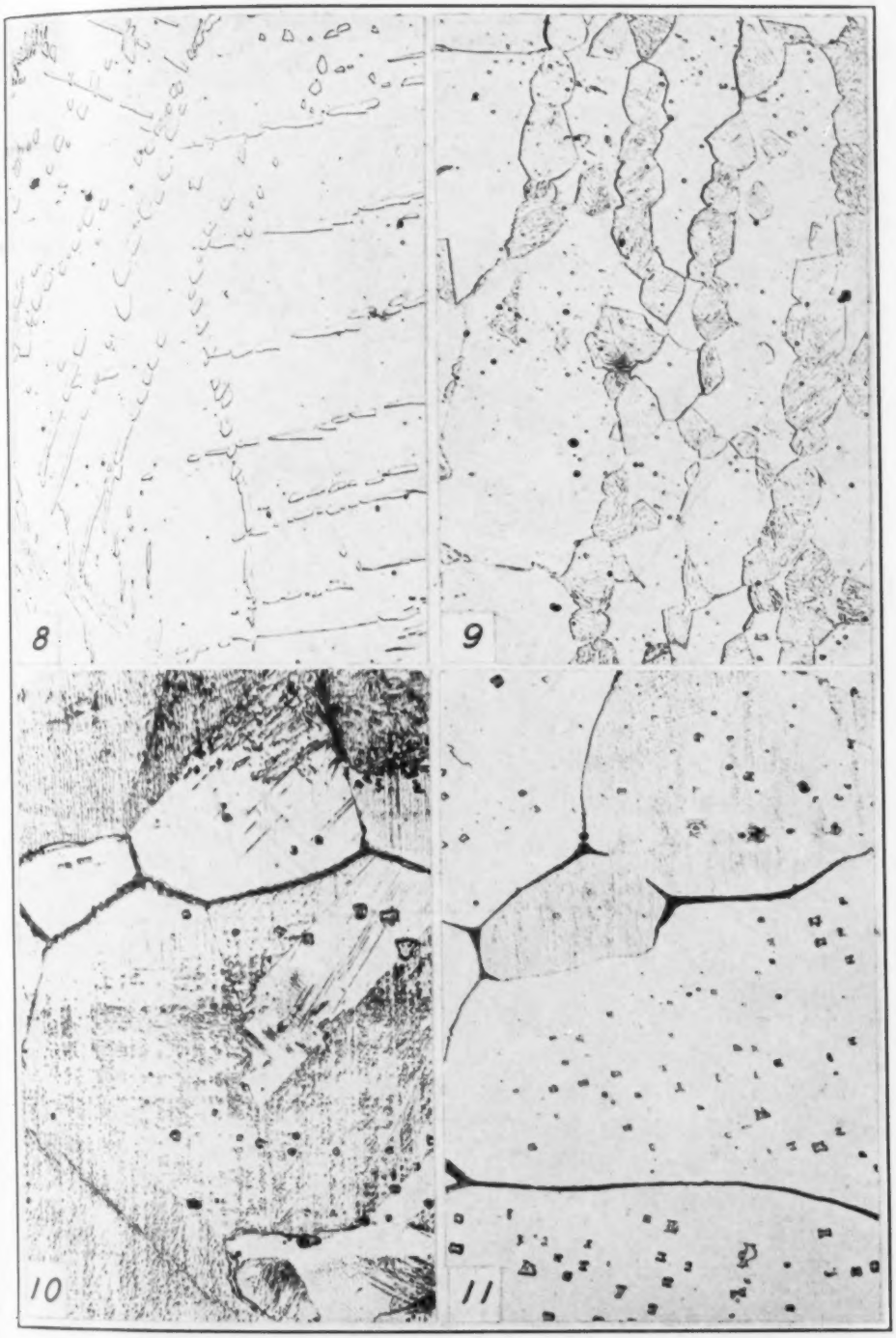


Fig. 8—Photomicrograph of Steel No. 205 at 2400 Degrees Fahr. Delta Ferrite with Austenite which Precipitated upon Cooling. Fig. 9—Steel No. 205 at 2300 Degrees Fahr. Grains of Austenite and Delta Ferrite in Equilibrium. Fig. 10—Alloy No. 234 at 2450 Degrees Fahr. Austenite partly Decomposed to Martensite, also Some Liquefied Metal. Fig. 11—Alloy No. 260 at 2450 Degrees Fahr. Austenite plus Liquefied Metal. All Photomicrographs × 250.

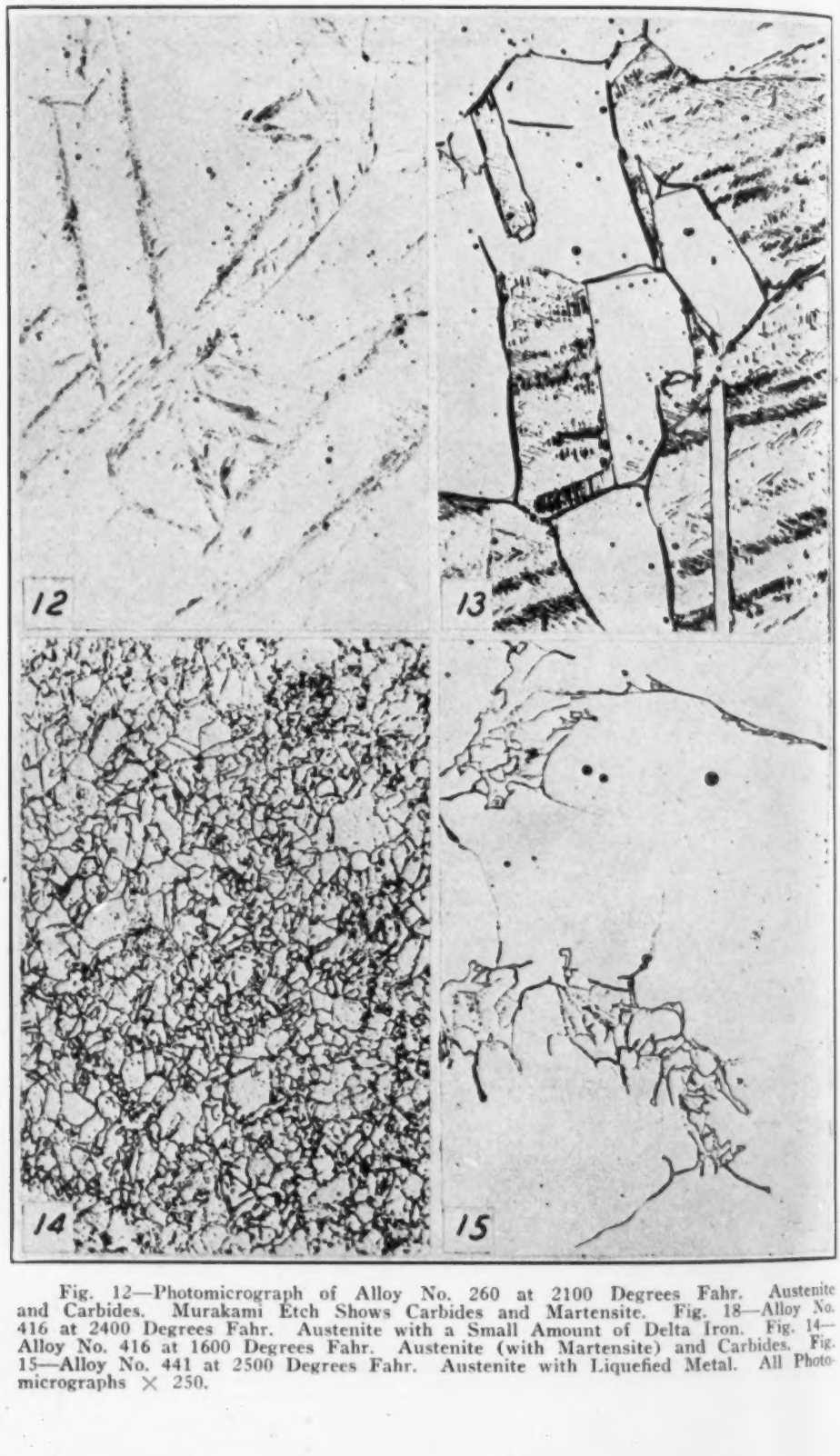


Fig. 12—Photomicrograph of Alloy No. 260 at 2100 Degrees Fahr. Austenite and Carbides. Murakami Etch Shows Carbides and Martensite. Fig. 18—Alloy No. 416 at 2400 Degrees Fahr. Austenite with a Small Amount of Delta Iron. Fig. 14—Alloy No. 416 at 1600 Degrees Fahr. Austenite (with Martensite) and Carbides. Fig. 15—Alloy No. 441 at 2500 Degrees Fahr. Austenite with Liquefied Metal. All Photomicrographs × 250.

Table V

Rockwell C Hardness and Magnetic Index after Heating at 800 to 1400° F. (the steel having been in the as-forged condition).

Steel No.	800°F. 100 hours	1000°F. 100 hours	1200°F. 50 hours	1400°F. 50 hours
037	37 M	34 M	27 M	17 M
045	39 M	31 M	27 M	19 M
205	32 M	19 M	20 M	22 M
243	30 NM	28 SM	27 QM	44 M
405	38 M	25 M	26 M	28 M
434	30 NM	32 SM	44 M	42 M
441	34 VSM	35 SM	46 M	46 M
823	30 NM	31 NM	35 SM	29 NM
834	29 NM	30 NM	33 SM	31 NM
1222 A	19 NM	18 NM	23 NM	17 NM
1222 B	20 NM	21 NM	26 NM	23 NM
1230	23 NM	26 NM	24 NM	24 NM
1243	30 NM	28 NM	38 NM	34 NM

In indicating steel, last two numbers are carbon content, these being preceded by nickel content. Thus No. 823 is 0.23 per cent carbon with 8 per cent nickel. Figures in columns are hardness Rockwell C. M = magnetic. QM = quite magnetic. SM = slightly magnetic. VSM = very slightly magnetic. NM = non-magnetic.

the agglomeration of minute precipitated carbides. A martensitic structure is present particularly in the alloys of low nickel content and low carbon content.

Where an increase in hardness is noted, this is to be attributed to decomposition of some of the austenite accompanied by precipitation of very minute carbide particles and the formation of fine-grained alpha iron. The decomposition of the austenite is indicated in certain characteristic changes in the microstructure, and these will be discussed in detail in connection with the changes in magnetic properties.

A few characteristic groups of microstructures will serve to illustrate the changes observed. Fig. 22 shows the decomposition of a martensitic structure in alloy No. 037, containing, in association with the usual chromium, 0.37 per cent carbon with only a negligible nickel content. Progressively higher heating temperatures cause only softening of this alloy. It is magnetic throughout.

Fig. 23 shows the effect of heating alloy No. 243, with 2 per cent nickel and 0.43 per cent carbon. The lower reheating temperatures cause a slight softening of the austenite. As the reheating temperature is raised the austenite is caused to decompose, the effect of the decomposition being observed (a) in the magnetism, since the specimens gradually become more and more magnetic, (b) in the microstructure, since there is gradually developed an almost complete

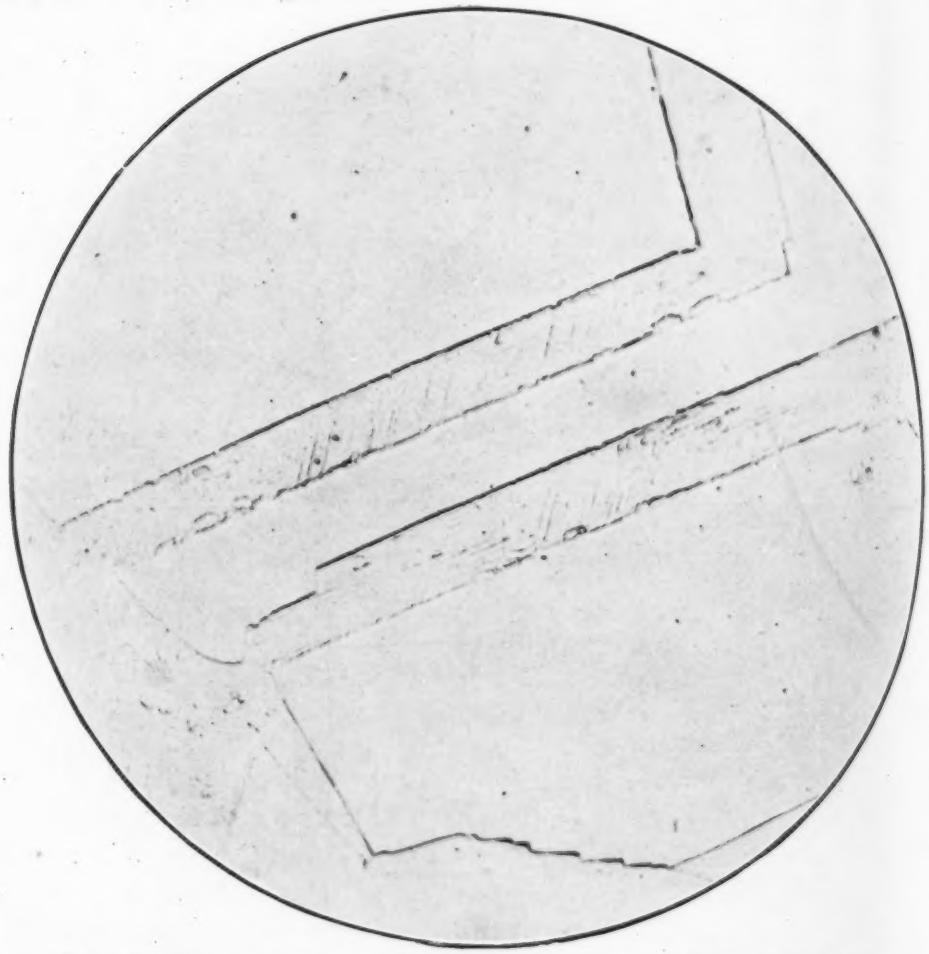


Fig. 16—Photomicrograph of Alloy No. 810 at 2100 Degrees Fahr. Austenite (Twins). × 2050.

precipitation of the carbides, and (c) in the hardness, since there is a very marked increase in hardness at the highest reheating temperature, 1400 degrees Fahr.

Fig. 24 shows the structures exhibited by alloy No. 434, with the usual chromium plus 4 per cent nickel and 0.34 per cent carbon. The structure is at first austenite, and this is still present after the heating at 800 degrees Fahr. At higher temperatures decomposition takes place, giving maximum hardness at 1200 degrees Fahr. At 1400 degrees Fahr. the hardness becomes less, and agglomeration of the carbides is much more distinctly visible. In conjunction with these manifestations of austenite decomposition, it is seen that the material changes from nonmagnetic to strongly magnetic.

Fig. 25 shows alloy No. 834, containing (besides the chromium)

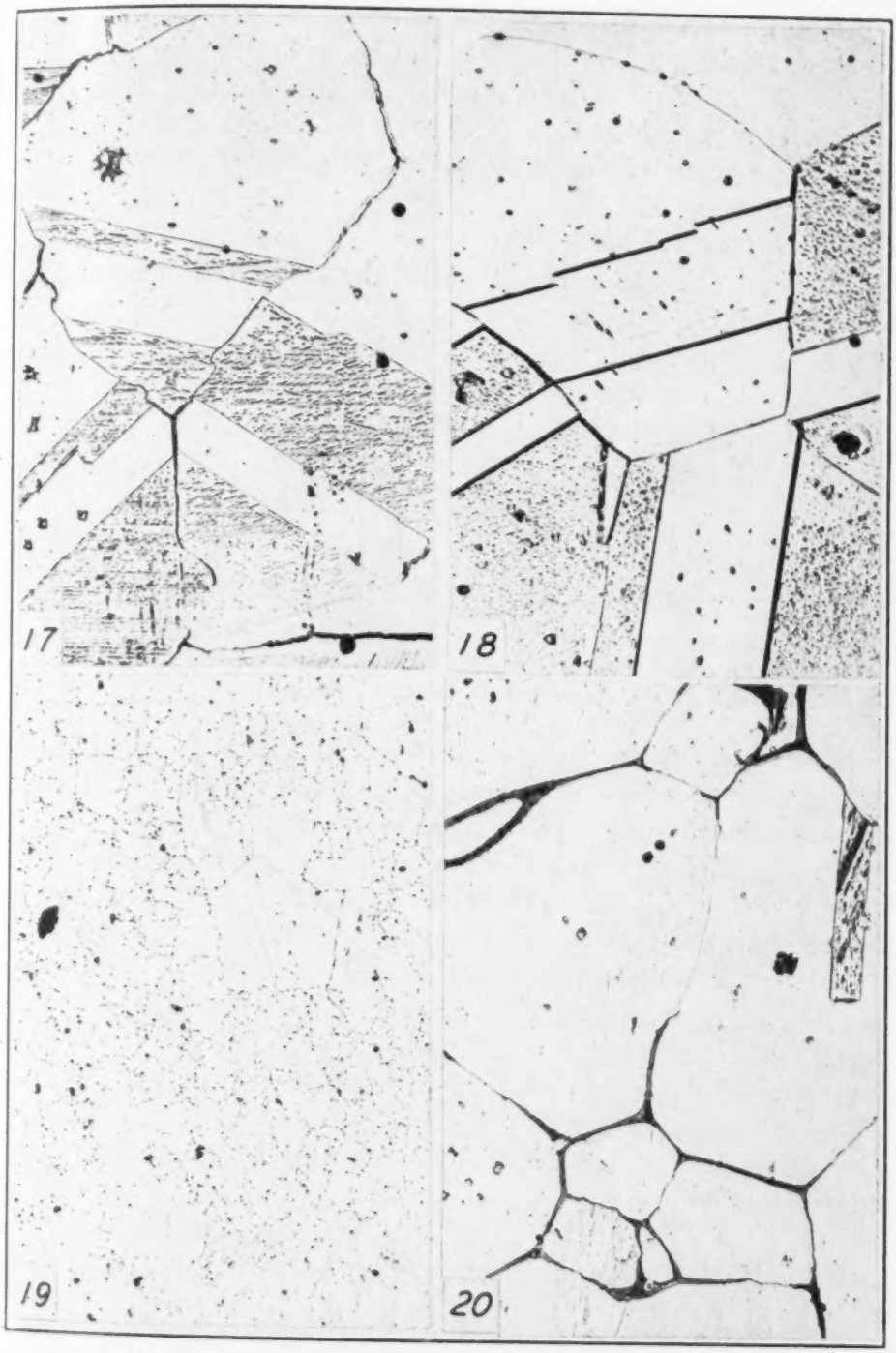


Fig. 17—Photomicrograph of Alloy No. 823 at 2450 Degrees Fahr. Austenite and Liquefied Metal. Fig. 18—Alloy No. 1231 at 2400 Degrees Fahr. Austenite. Fig. 19—Alloy No. 1231 at 1800 Degrees Fahr. Austenite and Carbides. Fig. 20—Alloy No. 1248 at 2450 Degrees Fahr. Austenite and Liquefied Metal. All Photomicrographs X 250.

8 per cent nickel and 0.34 per cent carbon. The austenitic structure is not appreciably affected by the 100 hours heating at 800 or 1000 degrees Fahr. At 1200 degrees Fahr. a change occurs, followed by another change at 1400 degrees Fahr. these changes being characteristic of the group of alloys containing 18 per cent chromium and 8 per cent nickel. The changes taking place in this temperature range will be examined in some detail.

It is seen that alloy No. 834 when heated at 1200 degrees Fahr. appears to precipitate minute carbides, it becomes slightly magnetic,

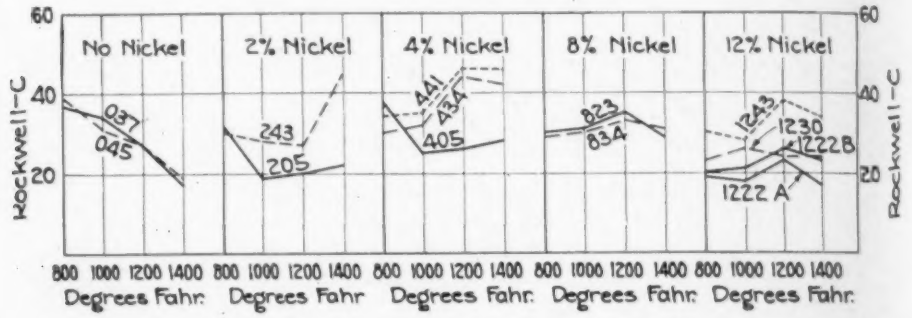


Fig. 21—Curves Showing the Effect on Rockwell C Hardness, of Reheating As-Forged Alloys in the Range 800 to 1400 Degrees Fahr.

and it increases somewhat in hardness. When heated at 1400 degrees Fahr. the carbides are rather more agglomerated, the steel is again nonmagnetic, and there is a decrease in hardness.

The precipitation of carbides upon heating at 1200 degrees Fahr. is illustrated in Fig. 26. This shows a specimen quenched from 2000 degree Fahr. and then reheated 250 hours at 1200 degrees Fahr., and etched with Murakami reagent to show the carbides. Now it is evident, upon reference to Fig. 5, that the reason for the carbide precipitation lies in the fact that these steels are hypereutectoid. The eutectoid point being below 0.05 per cent carbon at best, all steels containing more than 0.05 per cent carbon will precipitate carbide when heated in a suitable temperature range, below the hypereutectoid or Acm line.

It is not equally obvious why these steels should become magnetic in this temperature range, only to become nonmagnetic again upon heating to higher temperatures. A theory which has found general acceptance has, however, been published by Strauss and his co-workers Schottky and Hinnüber.[3] Their Fig. 16 is reproduced

[3]Strauss, Schottky and Hinnüber, "Die Carbidausscheidung beim Glühen von nichtrostendem unmagnetischem Chromnickelstahl", *Zeitschrift fur anorganische und allgemeine Chemie*, Vol. 188, 1930, pp. 309-324.

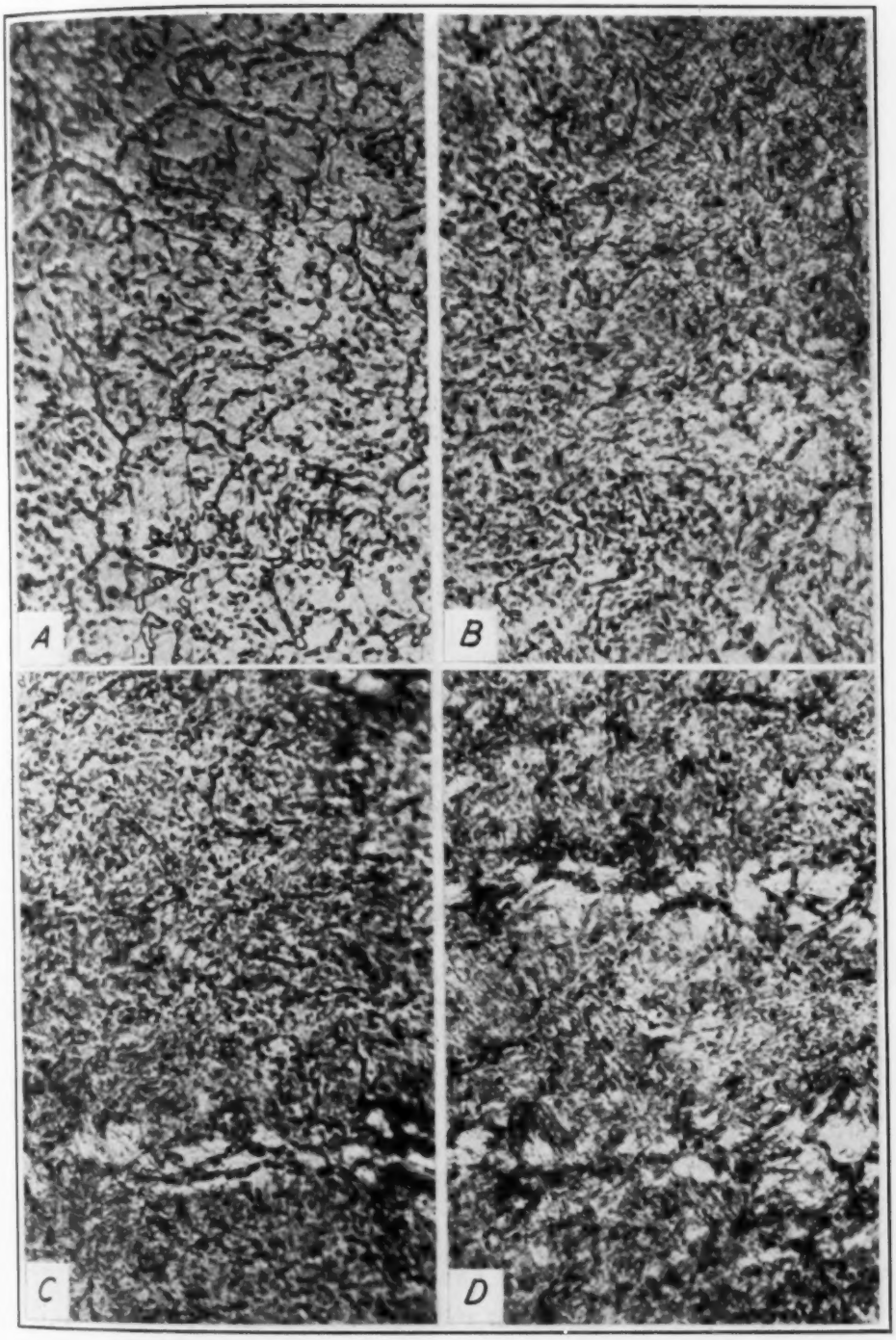


Fig. 22—Photomicrographs of Alloy No. 037 Reheated as Follows:
A. 1400 Degrees Fahr. Rockwell C Hardness 17.
B. 1200 Degrees Fahr. Rockwell C Hardness 27.
C. 1000 Degrees Fahr. Rockwell C Hardness 34.
D. 800 Degrees Fahr. Rockwell C Hardness 37.
All Specimens were Magnetic. Magnification × 1000.

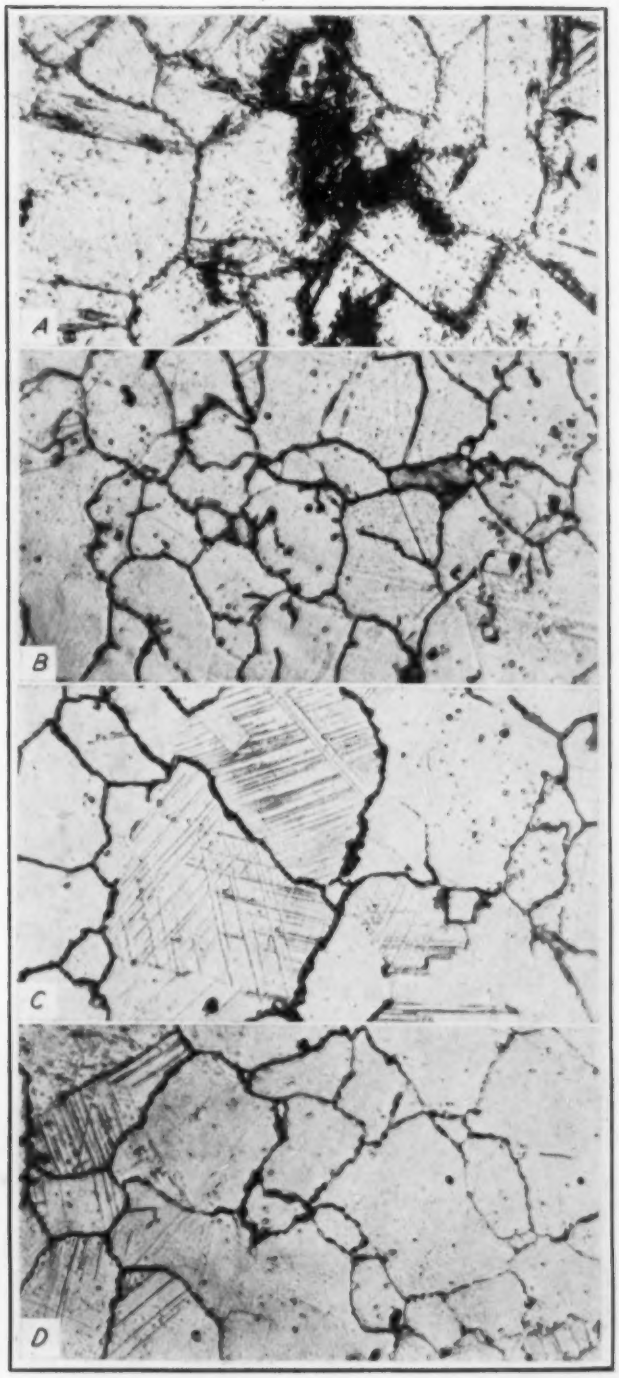


Fig. 23—Photomicrographs of Alloy No. 243 Reheated as Follows:
A. 1400 Degrees Fahr. Rockwell C Hardness 44. Magnetic.
B. 1200 Degrees Fahr. Rockwell C Hardness 27. Slightly Magnetic.
C. 1000 Degrees Fahr. Rockwell C Hardness 28. Very Slightly Magnetic.
D. 800 Degrees Fahr. Rockwell C Hardness 30. Nonmagnetic.
Magnification × 1000. Reduced Approximately One-Third in Printing.

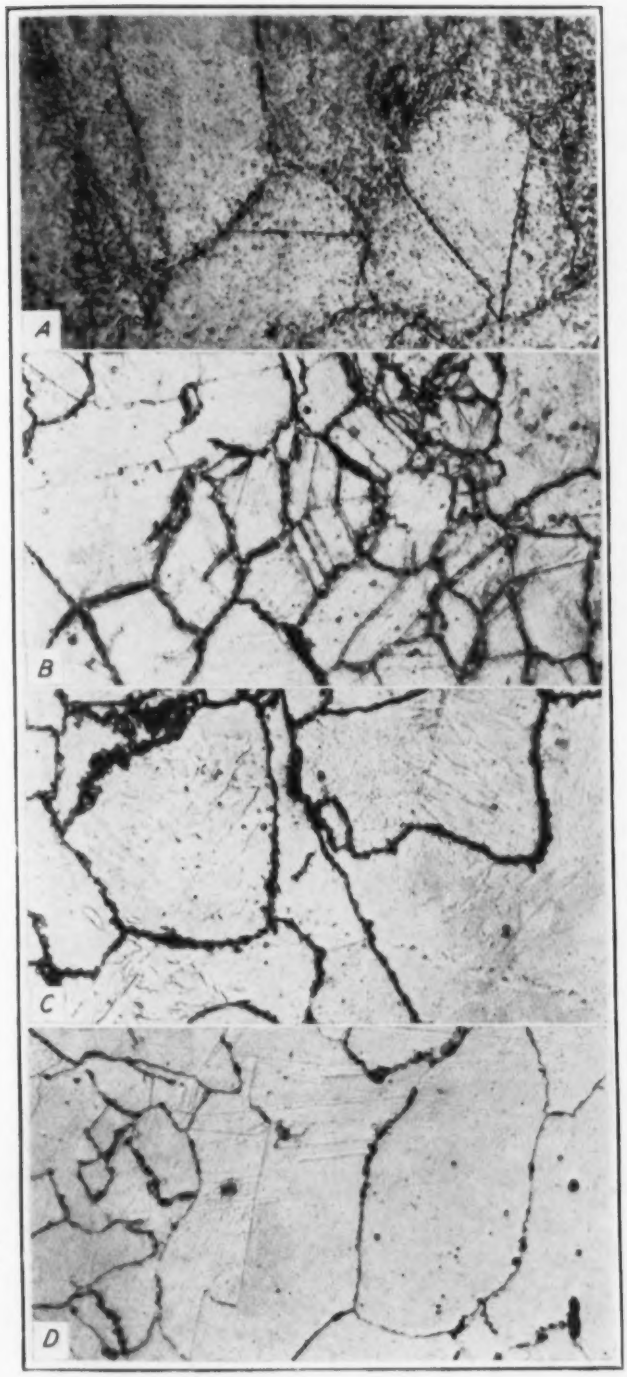


Fig. 24—Photomicrographs of Alloy No. 434 Reheated as Follows.
A. 1400 Degrees Fahr. Rockwell C Hardness 42. Magnetic.
B. 1200 Degrees Fahr. Rockwell C Hardness 44. Magnetic.
C. 1000 Degrees Fahr. Rockwell C Hardness 32. Slightly Magnetic.
D. 800 Degrees Fahr. Rockwell C Hardness 31-29. Nonmagnetic.
Magnification × 1000. Reduced Approximately One-Third in Printing.

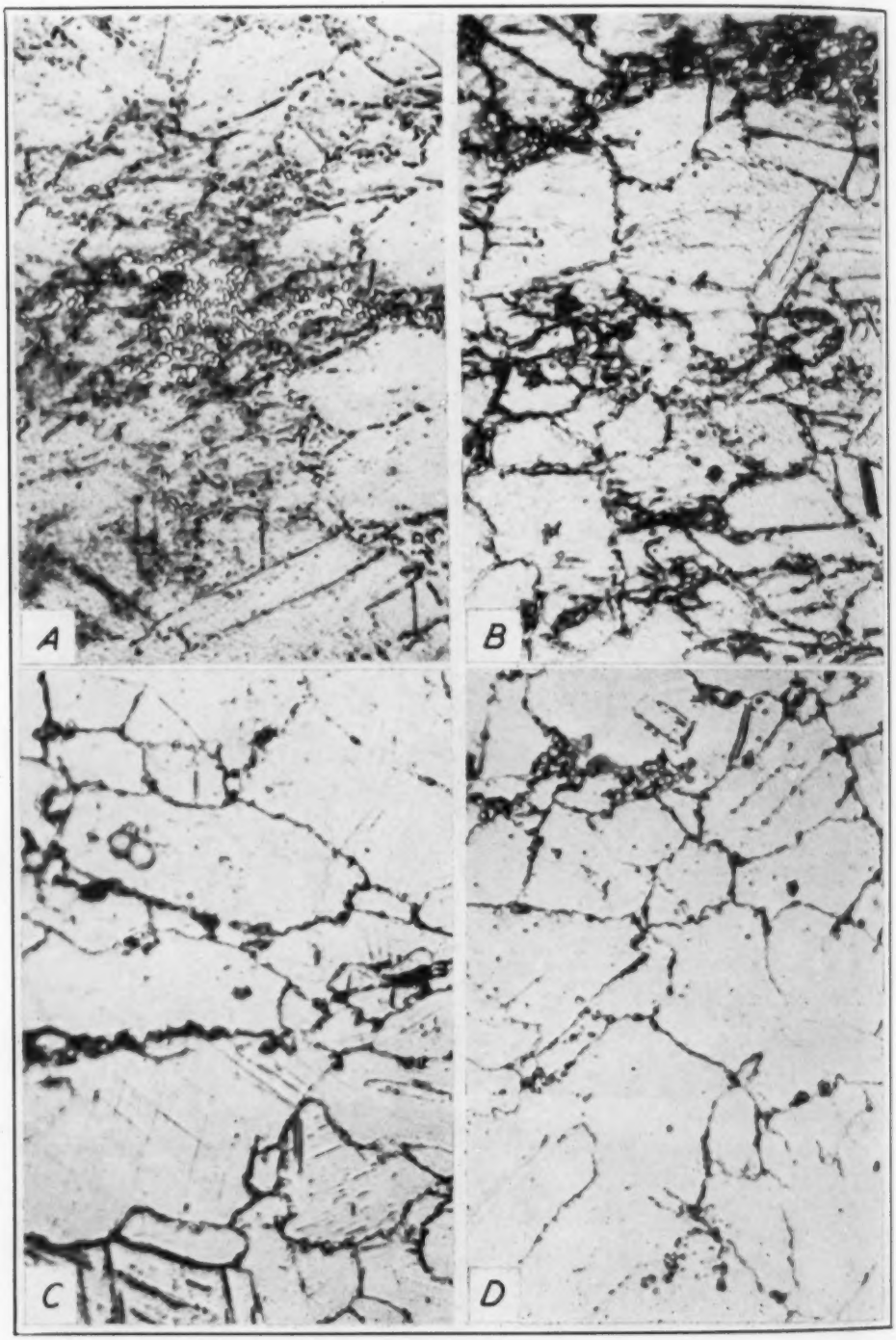


Fig. 25—Photomicrographs of Alloy No. 834 Reheated as Shown:
A. 1400 Degrees Fahr. Rockwell C Hardness 31. Nonmagnetic.
B. 1200 Degrees Fahr. Rockwell C Hardness 33. Slightly Magnetic.
C. 1000 Degrees Fahr. Rockwell C Hardness 30. Nonmagnetic.
D. 800 Degrees Fahr. Rockwell C Hardness 29. Nonmagnetic.
Magnification × 1000.

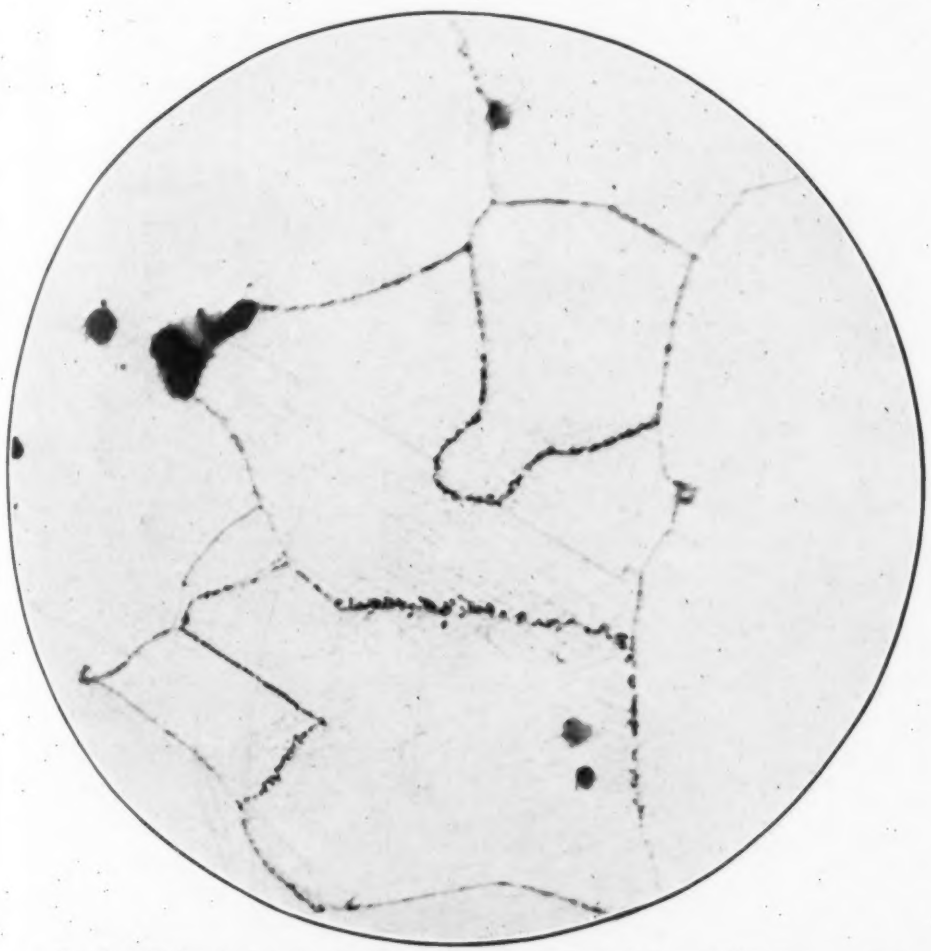


Fig. 26—Photomicrograph of Alloy No. 823, Quenched from 2200 Degrees Fahr. into Iced Brine, Reheated 250 Hours at 1200 Degrees Fahr. Murakami Etch. × 2050.

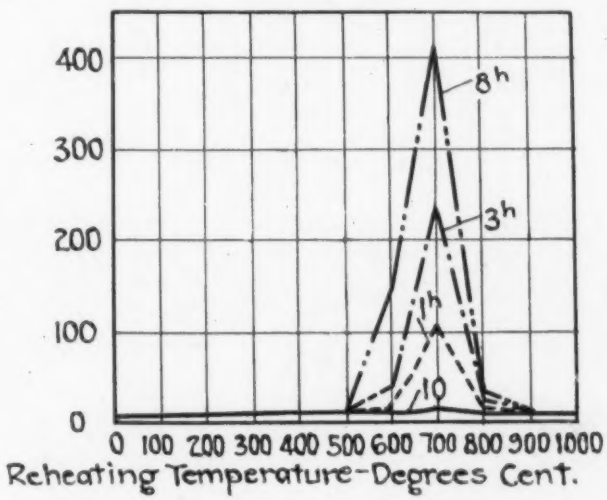
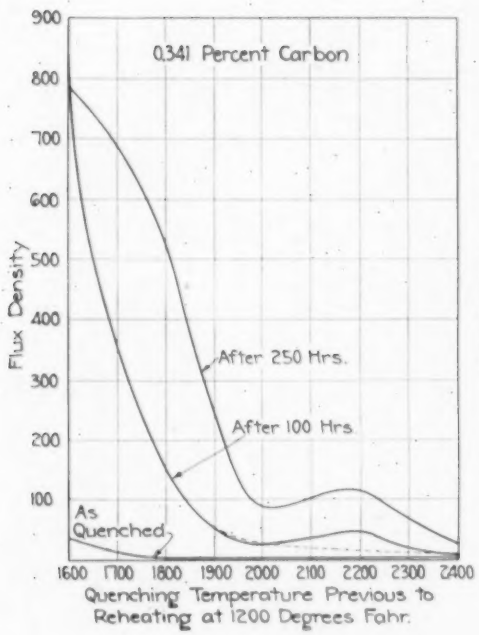


Fig. 27—Curves Showing Effect Upon Magnetic Properties, of Reheating at 700 Degrees Cent. (1300 Degrees Fahr.) of an Alloy Containing Carbon 0.12 Per Cent, Nickel 8.4 Per Cent, Chromium 18.6 Per Cent. (Strauss, Schottky, Hinnüber.)



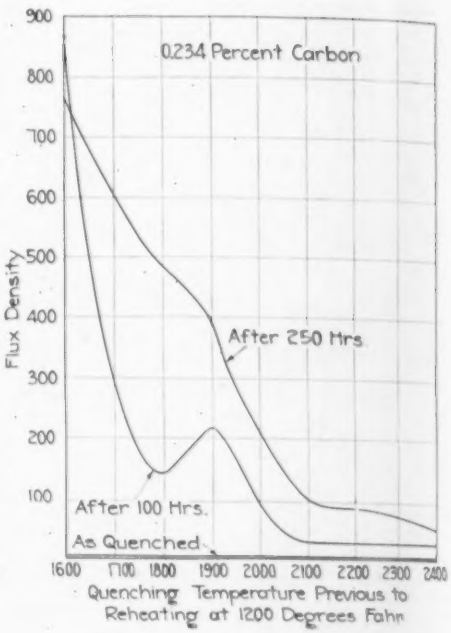
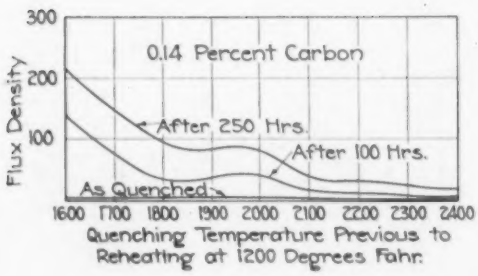


Fig. 28—Curves Showing Magnetic Properties of Alloy No. 834.
Fig. 29—Curves Showing Magnetic Properties of Alloy No. 823.



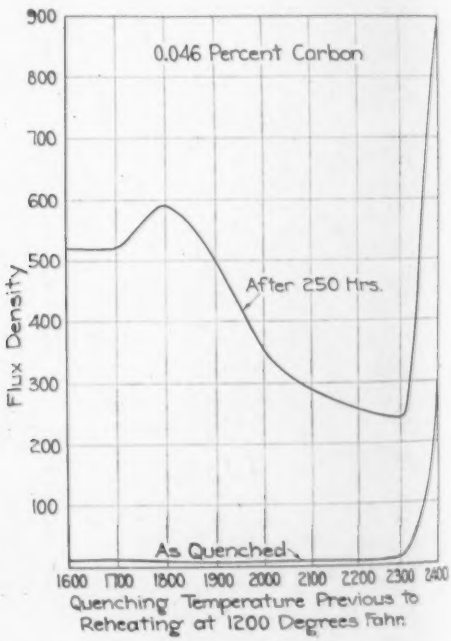


Fig. 30—Curves Showing Magnetic Properties of Alloy No. 814.
Fig. 31—Curves Showing Magnetic Properties of Alloy No. 804.

as Fig. 27 herewith. They show maximum magnetic flux after heating at about 1300 degrees Fahr. and explain it as follows. It

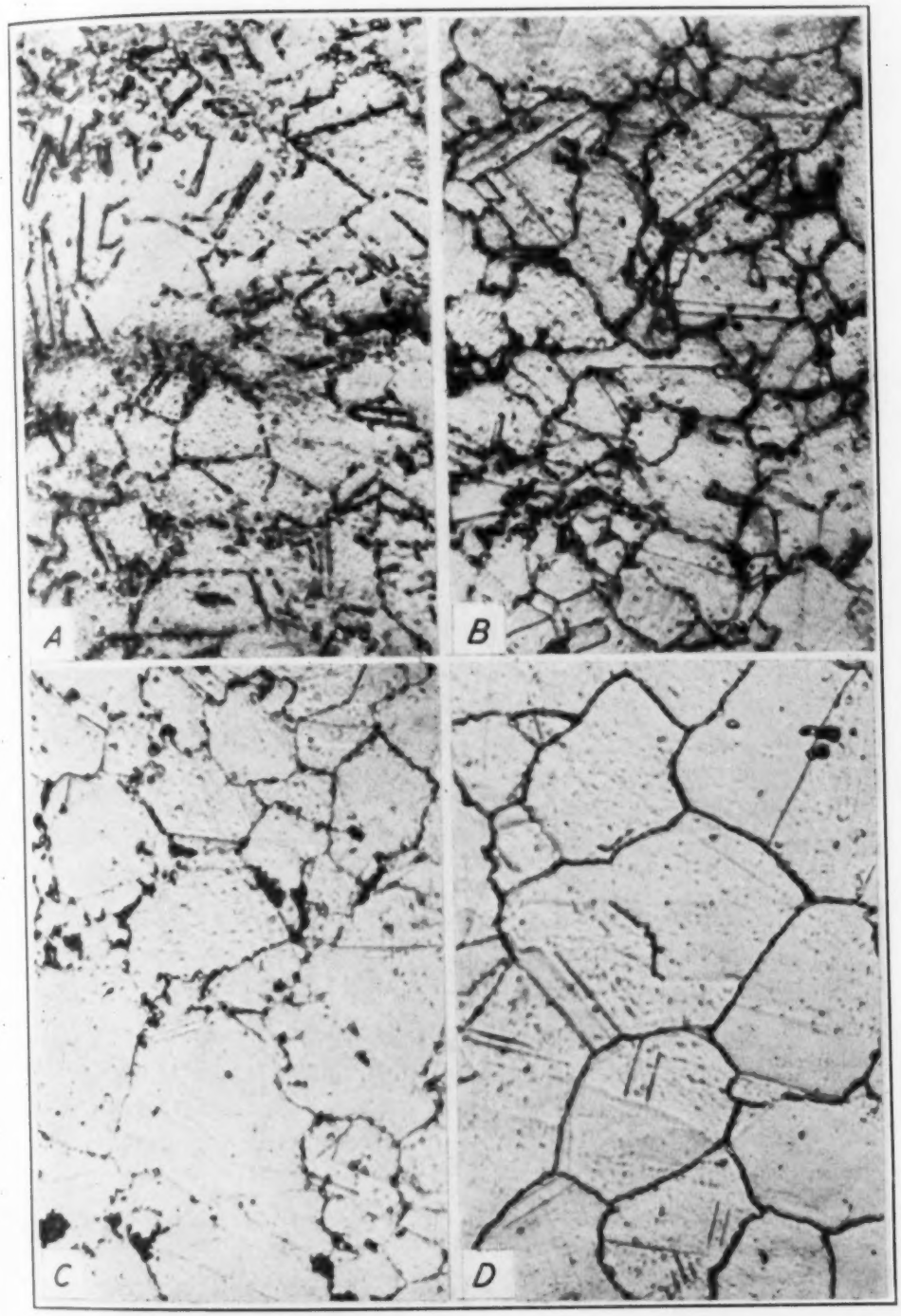


Fig. 32—Photomicrographs of Alloy No. 1222 Reheated as Follows:
A. 1400 Degrees Fahr. Rockwell C Hardness 23. Nonmagnetic.
B. 1200 Degrees Fahr. Rockwell C Hardness 25-26. Nonmagnetic.
C. 1000 Degrees Fahr. Rockwell C Hardness 21. Nonmagnetic.
D. 800 Degrees Fahr. Rockwell C Hardness 20. Nonmagnetic.
All Photomicrographs × 1000.

is well known that carbides in high-chromium steels contain a disproportionately large percentage of chromium. Thus the carbides in an 18 per cent chromium steel will usually contain 50 to 60 per cent chromium. This undue enrichment of chromium in the carbide leads to a corresponding impoverishment of chromium in the matrix immediately adjacent to the carbides, and this matrix being then poorer in alloy is no longer stable austenite in this temperature range. It changes at once to alpha ferrite, which is, of course, magnetic.

A series of magnetic tests was run on steels of this alloy content (18 per cent chromium and 8 per cent nickel), with various carbon contents. They were first quenched from a series of temperatures from 1600 to 2400 degrees Fahr., and then all reheated to 1200 degrees Fahr. for periods of 100 hours and 250 hours. The magnetic tests were carried out in a Fahy permeameter as follows:

The forged or hot rolled bars (about ⅝ inch round) were cut into 2¼ and 2½ inch lengths and annealed at 1800 degree Fahr. for one hour. Heat treatments varying from 2400 to 1600 degrees Fahr., ice quenched were then made. The 2400 and 2300 degrees Fahr. samples were held for ¼ hour at temperature, the others for ½ hour. This change in time was made to avoid grain growth and decarburization at the higher temperatures.

In order to avoid cold work the samples were not stamped to maintain their identity. Each specimen was wrapped in a slip of paper, and so carried throughout the tests.

The heat treated samples were then machined. About 3/16 inch was taken off the sides and ends of each sample on a lathe in order to get rid of decarburization. The last cuts on the lathe were made very fine in order to avoid excessive cold work. These specimens were then tested in the Fahy permeameter.

After a 100 hour treatment at 1200 degrees Fahr. the samples were pickled in aqua regia to take off the scale and decarburized area. They were then tested in the Fahy permeameter. After this, the samples were put in the furnace for an additional 150 hours, making a total of 250 hours at 1200 degrees Fahr. Microsections were cut after the 250 hours treatment, and magnetic tests were again run.

The magnetic data for the several carbon contents are assembled in Figs. 28, 29, 30 and 31. They show two definite trends: the lower the quenching temperature, the higher the flux density after reheating, and the higher the carbon content (shown also by Strauss,

Schottky and Hinnüber[5]) the higher likewise the flux density after reheating. That is, although all the steels are nonmagnetic after the quench, they show a greater susceptibility to the formation of alpha ferrite if the quenching temperature was low or if the carbon content was high. Neither of these laws holds, however, if the carbon content is as low as 0.04 per cent, with the alloy content as shown.

If the reheating temperature is raised to 1400 degres Fahr. the alloy again becomes practically nonmagnetic, and this may be due to a combination of causes. The steel is not so far hypereutectoid as it was at 1200 degrees Fahr. so that some of the carbide goes back into solution. Also the mobility of the atoms is greater, so that diffusion may cause an equalization of the alloy content. Further, the former alpha regions may enter the austenite range of temperature, depending on the alloy content.

Finally, we come to the 12 per cent nickel alloys, for which a typical series of low-temperaure heatings is shown in Fig. 32. It shows alloy No. 1222, containing 18 per cent chromium, 12 per cent nickel and 0.22 per cent carbon. The reheating temperature of 1200 degrees Fahr. is again typical in exhibiting precipitation of carbide and showing an increase in hardness. It is to be noted, however, that the alloy in this case remains nonmagnetic, due undoubtedly to the high nickel content which causes the areas around the precipitated carbides to remain austenitic in spite of the loss of chromium. The alloy remains nonmagnetic throughout the series of heating temperatures from 800 to 1400 degrees Fahr.

Summary

A series of alloys was examined, all containing 18 per cent chromium, but varying in nickel content from 2 to 12 per cent and in carbon content from 0.05 to 0.60 per cent. The alloys were heated at various temperatures from 800 to 2500 degrees Fahr. and examined for microstructure, magnetic properties and hardness.

Constitutional diagrams are offered for the range of temperature from 1600 to 2500 degrees Fahr. For lower temperatures, from 800 to 1400 degree Fahr., where heating fails to establish equilibirum, certain peculiarities of behavior are discussed.

Acknowledgments

We wish to express our appreciation of the very generous help

extended by Robert Tillman, Jr., Harry Schultz, B. N. Daniloff, Max W. Lightner and George P. Motok, formerly graduate students at Carnegie Institute of Technology whose assistance made possible the early completion of this paper. We are indebted also to H. J. Hand, E. L. Beardman and Alessandro Reggiorri, who carried out all the magnetic tests, and whose cooperation is greatly appreciated; to Dr. Franklin H. Allison, Crucible Steel Co. of America, for friendly cooperation and permission to carry out magnetic measurements; to E. C. Wright, Ellwood Works of National Tube Co., for kindly supplying three sets of heat treated samples; to Miss Zita Hill, Herbert F. Brown and George F. Martin who have borne the burden of preparation of the samples for microscopic examination, and to J. C. Joublanc, who made all of the experimental heats used in these tests.

DISCUSSION

This paper by Doctors Krivobok and Grossmann was presented at the convention by Dr. Krivobok. Directly following, Mr. E. C. Bain presented his paper written jointly with R. H. Aborn entitled "Nature of the Nickel-Chromium Rustless Steels." This paper is published on pages 837-873 of this issue of TRANSACTIONS. All discussion of both papers was presented after the second paper, and due to the occasional over-lapping of discussion because of the similarity of subject matter in the two papers the discussion of both papers follows the Aborn and Bain paper on page 873.

NATURE OF THE NICKEL-CHROMIUM RUSTLESS STEELS

By Robert H. Aborn and Edgar C. Bain

Abstract

This paper endeavors to describe the behavior of the austenitic rustless steels in terms of their relation to the iron-chromium-nickel system as modified by the carbon content with special reference to low-temperature reactions.

The austenitic nickel-chromium stainless alloys of the usual analyses are not wholly austenitic at temperatures near the melting point except the carbon be in excess of some 0.15 per cent or the nickel be materially increased in general accord with the diagrams shown. Increased chromium increases the tendency toward ferrite separation.

At low temperatures, too, the alloys, regardless of carbon content are also unstable except the nickel be present in very high proportion. The low-temperature instability is of two types; instability of carbide solution and of crystal form or allotropy. The restoration of the former is brought about by reheating, whereupon excess carbide precipitates. Cold work will tend to develop the ferrite stable at low temperatures.

The actual course of these reactions constitutes the essential information necessary to the manufacture and use of the alloys. Magnetic measurements, microscopic observations, X-ray crystal analysis, corrosion tests, and hardness determinations have supplied the information which has served as a consistent basis for the views set forth.

The ferrite developed by cold work possesses the composition of the alloy as a whole; its gradual disappearance, therefore, upon heating is a behavior properly plotted upon a plane two-dimensional diagram. This constitutes a metastable constitution diagram wherein the counterpart of the A_1 and A_3 points for hypoeutectoid steel may be entered.

The ferrite developed as an accompaniment to car-

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The authors are members of the society and are on the staff of the Research Laboratory of the United States Steel Corporation. Manuscript received August 5, 1930.

bide precipitation is of a composition which may vary from that of the alloy almost to pure iron and is therefore a behavior which is better described than graphically represented.

A third type of constitution diagram which would really constitute a section of the true equilibrium diagram would represent the carbide as fully precipitated at low temperatures. Such a diagram has been attempted from deductions from the partially metastable behavior.

In view of the unavoidable complexity of the structural changes in the system, a single alloy is finally followed through its various changes with increasing temperature. Except where necessary to establish the existence of a tendency high accuracy has been made a secondary consideration. The discovery of the trends of the reactions and properties, it is believed, is of greater practical importance at this time, if followed later by more precise determinations, than the precise estimation of isolated properties. Due to the length of the paper the details of the study of the individual alloys cannot be included.

Introduction

AMONG the iron alloys in which high corrosion resistance has been secured by adequate chromium addition, none, at the moment, is engaging greater attention than the nickel-chromium group in which the nickel addition is sufficient to render the material austenitic and non-magnetic; a typical composition contains about 18 per cent chromium and 8 per cent nickel. Chromium is the outstanding element of the group which confers the property of inert surface film formation upon its iron alloys; moreover it may be added in large proportions and still yield a malleable alloy. Nickel is a logical addition to the iron-chromium materials for it possesses, to some extent at least, two tendencies associated with potential corrosion resistance. In many aqueous environments nickel shows a more fundamentally noble behavior than iron, i. e., it has lower solution pressure; in addition, it seems to possess the ability to develop surface passivity, at least to some extent, in the manner of chromium and of the chromium-iron solid solutions.

That the addition of some 10 per cent nickel to the iron-chromium alloys should render them austenitic is of less advantage to the corrosion behavior than to the mechanical properties. The crystal structure, whether the alloys are austenitic or ferritic, depends

upon the temperature and heat treatment as well as upon composition. Some understanding of the general nature of the entire ternary series of alloys containing all possible combinations of iron, chromium and nickel is very helpful in gaining the necessary familiarity with the detailed behavior of this restricted group of compositions to insure proper treatment in manufacture and use.

It is the object of this paper to discuss the behavior of the so-called "18-8" alloy (and its neighbors in composition) particularly from the standpoint of its relation to the system as a whole.

Preliminary Discussion

The ternary system of alloys containing iron, chromium, and nickel has, even at first glance, interesting if not unique characteristics. Within some temperature range iron forms a complete unbroken solid solution series both with nickel and with chromium, but these solid solutions differ. Nickel possesses only the crystal form of gamma iron and forms accordingly a complete and unbroken solid solution with it in all proportions. Chromium exists only in the crystal form of alpha (and delta[1]) iron and forms with it a solid solution in all proportions. The solid solutions of the gamma iron-nickel type of crystal (face-centered cubic) are here designated as austenite while those of the alpha iron-chromium type (body-centered cubic) are here termed ferrite. The ability of iron to exist in each of its two forms over certain temperature ranges causes interesting changes in the crystal forms of all the iron-rich alloys. Both at low and at extremely high temperatures the alpha iron crystal form or ferrite predominates and so exists even with considerable nickel dissolved while at intermediate temperatures—between 900 and 1400 degrees Cent. (1650 and 2550 degrees Fahr.)—the austenite form is dominant, and this gamma iron crystal type persists even with considerable chromium present.

In the alloys of chromium and nickel alone (without iron) only these same types of solid solution may exist. Nishigori and Hama-

[1]The structural identity of alpha iron and delta iron is now so well established and in the iron-chromium alloys they are so completely merged that the word "delta" will not be used again. "Ferrite" will designate all solid solutions of alpha iron, and "austenite" will designate all solid solutions of gamma iron. The use of "nickel-austenite" or "chrome-ferrite" would, in this discussion, only be redundant and tedious. How near to pure chromium compositions may approach and still be termed "ferrite" is a matter of the reader's opinion and as no ambiguity is encountered, it is left to his choice.

sumi[2] very recently indicate that at the eutectic temperature nickel may dissolve (in solid solution) about 46 per cent chromium while chromium may dissolve as much as 36 per cent nickel. The intermediate compositions would be a mixture of the two types of solid solution (body centered and face centered cubic), the one corresponding in crystallinity to, and miscible with the already mentioned austenite and the other similarly isomorphous with ferrite. At low temperatures the range of two-constituent mixtures widens so that only 7 or 8 per cent nickel is dissolved in chromium and 35 per cent chromium dissolves in nickel. This nickel-chromium diagram agrees qualitatively (and to a first approximation quantitatively) with that proposed by one of the present authors in 1923. It should be possible then to construct an approximate ternary constitutional diagram at once since only two kinds of solid solutions exist in the entire iron-chromium-nickel system. A field for each and a heterogeneous field is all that can be encountered[3] and the equilateral diagram, constructed for any temperature level will have very simple characteristics.

The Ternary Diagrams. Obviously, the construction of a precise ternary equilibrium diagram for the complete significant temperature range is a task of gigantic proportions. However, the greatest usefulness of the ternary diagram is in indicating the trends which may be expected in individual compositions, and for this purpose the highest accuracy is not required. It is believed, for reasons which will later appear, that some working hypothesis for the general behavior of a considerable range of ternary compositions must usually be drawn up if one is to study efficiently a single composition or narrow range within the group. A single ternary composition in a range of possible heterogeneity is almost certain to develop phases with composition not in any single plane of intersection and, except a solid diagram of trends be sketched at the inauguration of study, the interpretation is likely to be difficult or ambiguous.

The authors, in providing such a preliminary three-dimensional chart, have drawn information from all possible sources in addition

[2]S. Nishigori and M. Hamasumi, The Equilibrium Diagram of Nickel and Chromium, Sci. Repts. Tôhoku Imperial Univ. Ser. I. Vol. 18, 1929, p. 491.

[3]The brittle constituent discovered by Bain and Griffiths may be omitted since it is not always developed. This brittle constituent may, further, require nitrogen, so readily picked up by the alloys, for its formation. Whether, as in the gold-copper series, a compound may form at lowest temperature for diffusion in the iron-chromium series is still unsettled. It would probably have the formula FeCr with nickel substituting in part for iron.

to their own studies. In the main, they have had to attempt to adjust the work of earlier investigators whose alloys contained variable carbon, to approximate negligible carbon content; this has been done partly by carefully estimating the carbon effect in a few alloys and modifying the results accordingly. Fortunately they have had available a few compositions of exceedingly low carbon content (0.02 and 0.03 per cent) in the composition range of particular interest.

The outstanding property of the alloys containing substantial

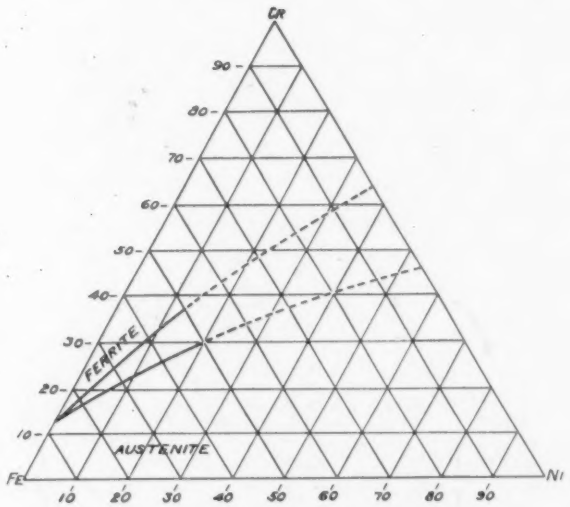


Fig. 1—Division of the Iron-Nickel-Chromium System into the Ferrite, Austenite and Mixed Fields as Occurring at Temperatures of Maximum Austenite.

proportions of chromium and nickel is their sluggishness toward all action involving diffusion and change of phase at all moderate temperatures. For example, the "18-8" alloy itself is truly stable only as ferrite at temperatures below about 400 degrees Cent. (750 degrees Fahr.) but indefinite maintenance at this temperature will not in itself cause the alloy, as quenched in the austenitic condition, to transform from metastable austenite to stable ferrite. Cold work, however, supplies the necessary atomic "stirring" to induce the alloy, at least partially, to assume its stable condition. The word "stable" is here used in its physical-chemical sense, and not, as occasionally found, in the sense of "persistent."

Before discussing the results of study of the restricted field under consideration a set of ternary diagrams corresponding to various temperature zones may be considered. These have been constructed from the most recent information available and are probably

fairly accurate in the region of intensive study. Fig. 1 shows the division of the system into the three fields mentioned above as found at the temperature range (varying for different compositions) at which austenite extends over the widest composition range. At the chromium-nickel side of the diagram the term austenite is used (with realization of its possible mis-application) for the nickel-gamma iron type of crystal which is isomorphous with gamma iron. At this region the widest austenite extension is at the incipient melting point.

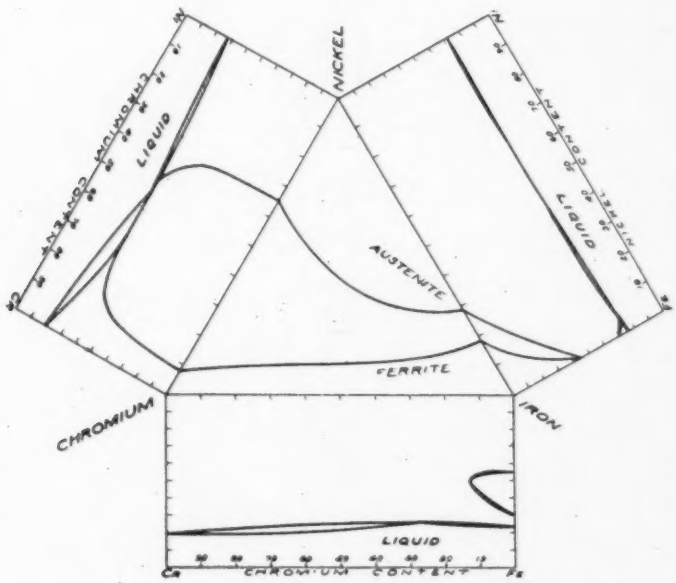


Fig. 2—Division of the Iron-Nickel-Chromium System into the Ferrite, Austenite and Mixed Fields as Probably Existing in Equilibrium at Room Temperature. Binaries are Appended.

Fig. 2 is an approximate diagram indicating the extension of the three fields as probably existing at equilibrium at or about room temperature. By thermal agencies alone this condition could obviously be brought about with any certainty only at much higher temperature, but the effect of cold work to show the trends of equilibrium points to some such state as indicated. The super-position of Figs. 1 and 2 outlines the greatest composition range of possibly heterogeneous alloys, which would consist of both ferrite and austenite. Due to the influence of the high-temperature alpha iron at temperatures above those shown in Fig. 1 (except near the nickel-chromium edge) the ferrite type of solid solution increases in composition range toward higher nickel just below the temperature of incipient melting.

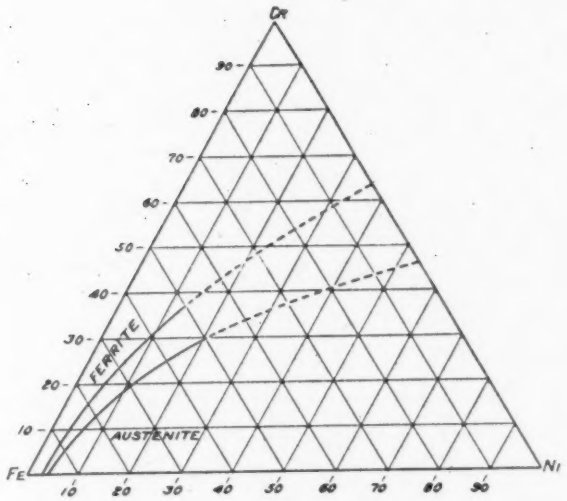


Fig. 3—Division of the Iron-Nickel-Chromium System into Ferrite, Austenite and Mixed Fields at Temperatures near Incipient Melting.

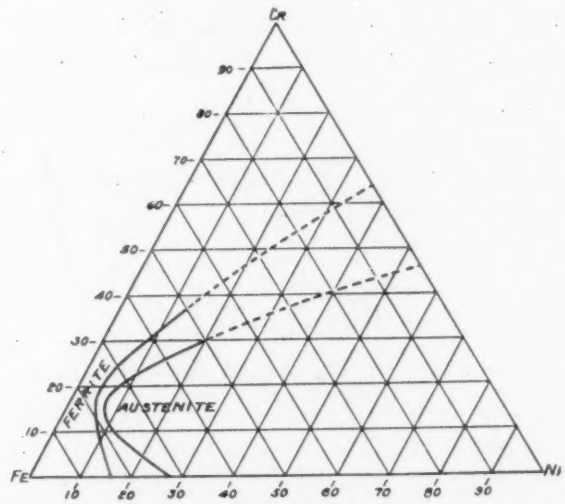


Fig. 4—Division of the Iron-Nickel-Chromium System into the Ferrite, Austenite and Mixed Fields as Developed by Rapid Cooling from Temperatures of Fig. 1.

Fig. 3 shows the three fields at this high temperature. It will be noted that here again, as in the low-temperature chart of Fig. 2, the ferrite form extends fully across the diagram into the iron-nickel binary. Fig. 4 shows the fields resulting from rapidly cooling the alloys to room temperature from the temperature range of Fig. 1. It will be noted here that chromium (and chromium with nickel) contributes such sluggishness to the alloy that many compositions, which would be in accordance with equilibrium change from the austenitic

to the ferritic state, are unable to undergo transformation. For the purposes of this estimate no distinction is made between polyhedral ferrite and ferrite with martensitic characteristics. Obviously the precise location of the fields as actually observed varies with cooling rate.

In order to illustrate further the outstanding tendency of the iron-rich alloys to become wholly austenitic at temperatures in the vicinity of 1000 to 1200 degrees Cent. (1825 to 2200 degrees Fahr.) a portion of the solid ternary diagram is shown in Fig. 5 with chromium and nickel content plotted on the horizontal plane and temperature on the vertical axis. The drawing is in isometric projection with four vertical planes constructed for a uniform chromium content of zero, 9, 18, and 24 per cent.

Another method of regarding these planes in the solid diagram is as constant-chromium sections plotted on oblique coordinates; the abscissa being increasing nickel and the ordinate temperature. Fig. 6 shows the phases developed in the 18 per cent chromium-variable nickel alloys and in the 8 per cent nickel-variable chromium alloys. The former is essentially identical with one of the sections of the solid diagram.

It will be noted in Fig. 5 that the alloys which are rich in iron are all shown with the peritectic type of solidus-liquidus combination as exists for the iron-nickel and iron-carbon systems. It seems well established that this is correct, indeed, the nickel rustless irons are a particularly representative example of this solidifying behavior. In a word, the solidification of these alloys proceeds as follows. The first crystallites to separate from the melt are ferrite, richer in iron than the melt. When the cooling continues, further ferrite is crystallized out upon the first crystallites but of composition more nearly that of the original melt. When a certain temperature, known as the peritectic, is reached the already solidified ferrite reacts with the liquid and (partially or completely depending on the total composition) changes to austenite and the remaining melt solidifies as austenite. In ingots of suitable composition these regions of ferrite are generally in evidence, especially near the surface. The subsequent transformation of the thus solidified ferrite to austenite at lower temperatures will be discussed further on.

From the foregoing summary of the entire iron-nickel-chromium system it may be seen that the alloys in the range of composition in the vicinity of 18 per cent chromium and 8 or 9 per cent nickel are

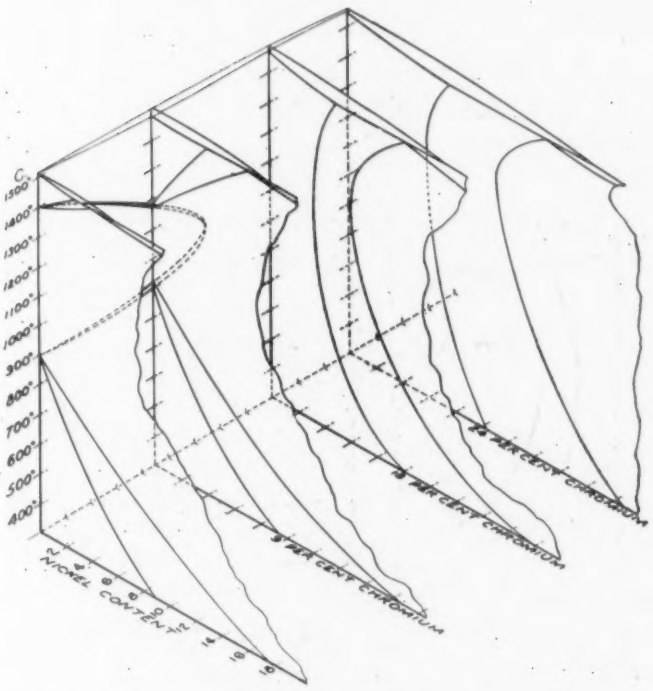


Fig. 5—The Approximate Equilibrium Diagram for the Iron-rich Nickel-Chromium Alloys. Four Uniform Chromium Sections are Shown.

subject to a tendency toward fairly involved transformations. It may be seen that higher chromium increases the tendency for high-temperature ferrite to form and for low-temperature ferrite as well, except that by its sluggishness it makes equilibrium less likely to be attained. Nickel, on the other hand, widens the temperature range of austenite stability and thereby lessens the tendency for either the high-temperature ferrite to appear or the low-temperature ferrite to be developed. With about 11 per cent or more nickel present an alloy of 18 per cent chromium will show no ferrite separation after heating just under the peritectic temperature at about 1410 degrees Cent. (2570 degrees Fahr.).

The Carbon Effect. The foregoing paragraphs have dealt with the behavior of the iron-chromium-nickel alloys without the consideration of the complicating influences of carbon. With a fair degree of success it is possible to plot a single behavior or property against composition if the composition involves only two components, or three, if the three comprise the whole or 100 per cent. Obviously, however, four dimensions cannot even be represented by any kind of solid chart, much less be represented on a printed page. The graphical

method then becomes impracticable for presenting any properties of the iron-carbon-nickel-chromium system as a whole. One may, however, fall back upon the admittedly imperfect method of charting behaviors of a *single* iron-chromium-nickel composition as modified by

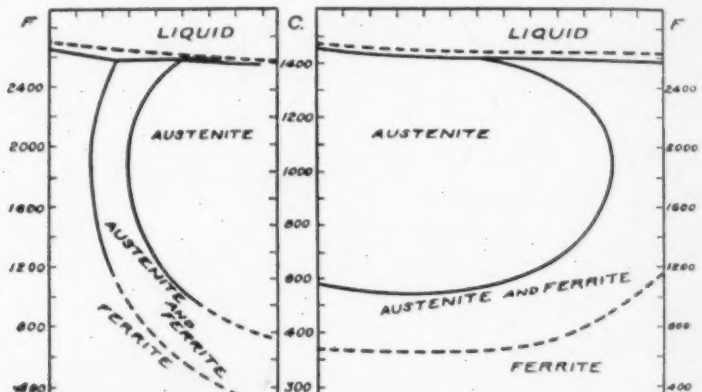


Fig. 6—The Approximate Constitution Diagram of the 18 per cent Chromium Alloys with Variable Nickel and the 8 per cent Nickel Alloys with Variable Chromium.

carbon content. This method has been employed in the following discussion of the alloys in the vicinity of the popular "18 and 8" material.

The effect of carbon upon the properties of the nickel rustless iron is very profound. Its solubility varies unusually with temperature; its concentration affects the separation of high-temperature ferrite and the lower transformation from ferrite to austenite upon heating. Finally, the mechanism of carbide precipitation changes completely the corrosion behavior of the alloy. It may be said that all the important metallurgical behaviors of the alloys are dependent upon carbon content and the main section of this paper treats of these properties particularly as affected by carbon content.

The Properties of the Nickel-Chromium Rustless Steels as Influenced by Carbon

Due to the sluggishness of the alloys of iron and chromium they may easily exist at a low temperature with a constitution which is only really stable at elevated temperature, but which persists with

moderate cooling rate down to temperatures at which readjustment in conformity with equilibrium cannot take place. The austenitic alloys of nickel and chromium of the type under discussion are particularly subject to this sort of undercooling. As has already been mentioned in the introduction, the allotropic change may easily be avoided in the cooling, indeed it seems likely that, except for a unique carbon effect, the slowest rates of cooling will not permit the allotropic change of austenite to ferrite to occur. Moreover, these alloys when carrying a small carbon content may also be undercooled in respect to carbon solubility. Almost the whole of the interesting metallography of the materials may be explained on the basis of these two types of undercooling.

Carbide Solubility. If the carbon is wholly dissolved at elevated temperature, only very slow cooling to some lower temperature, at which the solution would be supersaturated, causes precipitation of the excess. The carbon in excess of that which may exist in solid solution is precipitated at a rate which depends almost wholly upon the temperature of the precipitation. Accordingly, to precipitate the carbide it is advisable to cool at a decreasing rate such as will just permit the precipitation to occur in accordance with the diminishing solubility. This is an exceedingly slow process because, while the excess carbide separates rapidly enough at high temperatures of rapid carbon (and chromium) diffusion, it may do so only at a very slow rate at low temperatures. Here then is a typical example of particle precipitation wherein a compound forms from a solid solution. In order to determine the solubility of carbon at various temperatures it has been found advantageous (after a few preliminary explorations) to use a method involving the *solution* rather than the *precipitation* as the indicator. The method has for its advantage the circumstance of requiring only a slight diffusion of elements to accomplish the complete change from austenite plus carbide to pure austenite.

The method is essentially as follows:

1. Heat to a temperature near that for ferrite separation (if any) to dissolve all carbon and to homogenize the alloy.

2. Reheat at a very low temperature just sufficient to precipitate sufficient carbide to serve as an indicator and still to preserve as far as possible the homogeneity of the alloy.

3. Heat rapidly to a variety of elevated temperatures at narrow

intervals and maintain at constant temperature until equilibrium results.

4. Examine microscopically for excess carbide particles and locate temperature of complete carbon solubility for each composition between the indicated two temperatures.

The alternate method of heating for complete solution and then

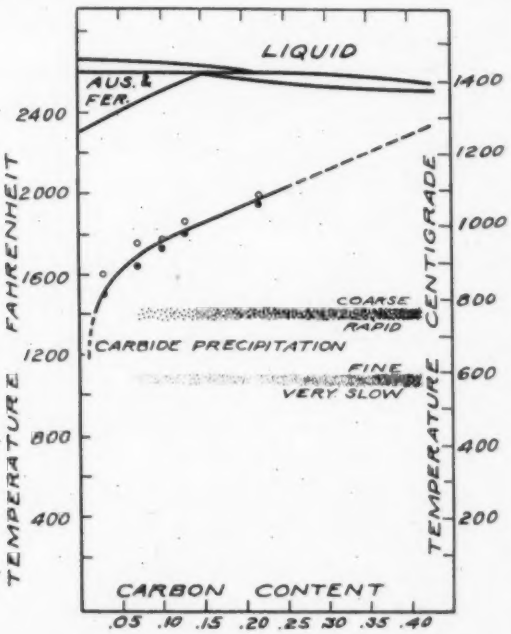


Fig. 7—The Solubility of Carbon in the Austenite of an 18 Chromium-8 Nickel Alloy.

dropping the temperature successively followed by examination of the quenched specimen is substantially the same except that the carbide precipitated at low temperature seems to be more easily observed.

In general, all specimens used in solubility determinations were maintained at temperature sufficiently long to show no further change by increased time. The necessary time was found to range from a few minutes at the highest temperature up to hundreds of hours at the lower temperatures.

The results of such solubility measurements are plotted graphically in Fig. 7. This chart shows the summary of the results more clearly than any description. The actual manner of the carbide precipitation will be discussed under a subsequent heading. It will be seen that the solubility of carbon increases very rapidly with temperature above 900 degrees Cent. (1650 degrees Fahr.). At lower tem-

peratures the solubility decreases less rapidly with falling temperature. Strauss[4] implies that the solubility of carbon is at least 0.04 per cent at about 600 degrees Cent. (1110 degrees Fahr.) but the present study suggests a slightly lower solubility. The solubility at high temperature is determined with somewhat greater accuracy than at low temperatures and in all probability the figure for solubility below about 550 degrees Cent. (1020 degrees Fahr.) must remain undetermined, the rate of diffusion of the carbide elements being too slow to establish equilibrium in reasonable time.

Another more serious source of vagueness in the solubility of carbon at low temperature lies in the fact that the carbide particles are not usually in equilibrium with austenite after precipitation but are surrounded by ferrite. Such ferrite might even be leaner in nickel than the remainder of the alloy. The carbide formed appears to be very rich in chromium and the adjacent metal probably is principally an iron-nickel alloy. Whether or not homogeneity is re-established just at the moment of re-solution is a matter for conjecture. At any rate there are a number of circumstances serving to cast doubt on the possibility, even, of determining the precise solubility of carbon at these low temperatures. In general these difficulties would tend to make the apparent solubility higher, rather than lower, than the true solubility.

The Allotropic Transformations. A. High Temperature

As already pointed out, the alloys containing comparatively small amounts of nickel or chromium in iron still exhibit the so-called "delta iron" influence and accordingly partly transform in the vicinity of 1300 to 1400 degrees Cent. (2370 to 2550 degrees Fahr.) from austenite to ferrite upon heating. Alloys of iron with nickel alone up to about 4 per cent nickel react in this manner, nickel in excess of this proportion permits only austenite up to the melting point, while the iron-chromium alloys exhibit this property even up to about 13 per cent chromium. With more chromium the ferrite exists at all temperatures and hence there is no austenite to transform. In a manner similar to the action of nickel the iron-carbon alloys are found to transform to ferrite in this temperature range but carbon in excess of about 0.2 per cent permits of no ferrite separa-

[4]Dr. Benno Strauss, H. Schottky and J. Hinnüber, Die Carbidausscheidung beim Glühen von nichtrostendem unmagnetischen Chromnickelstahl, *Zeit. für Anorganische u. Allgemeine Chemie*, 1930, Band 188.

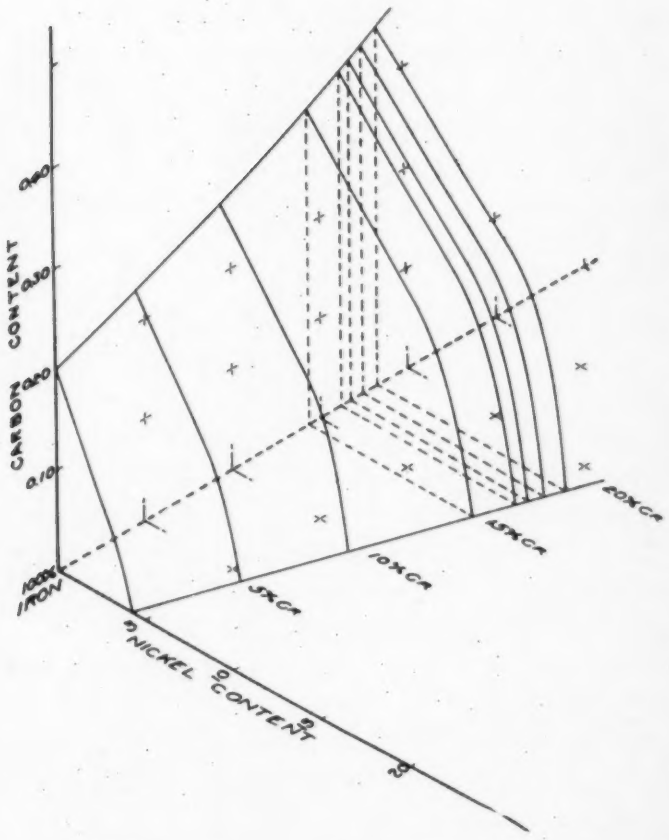


Fig. 8—Diagram Showing Probable Limits of Composition (Ni, Cr and C) of Alloys Developing High-Temperature Ferrite.

tion. Thus with the limits of carbon and nickel for ferrite separation well known and some fairly reliable information on the limits in the various ternary systems also available, the possibility exists of making a chart showing the limits of all three elements—chromium, nickel, and carbon—which will just permit high temperature ferrite separation. It has been found that chromium strengthens the tendency towards such separation while carbon and nickel diminish it. After examining about twenty alloys with nickel from 7 to 13 per cent, chromium ranging from 13 to 19 per cent and carbon from 0.02 to 0.22 per cent, the location of the limiting compositions in that range was fairly well established. The information available, when graphically indicated, results in the limit surface of Fig. 8.[5] This

[5]Since the conclusion of this work a private communication from Dr. M. A. Grossmann indicates definite although not serious differences from the values plotted. His observations are very comprehensive.

again is an isometric projection of a solid in which the customary x, y, and z axes are respectively nickel, chromium, and carbon content (iron = 100 per cent — [x + y + z]) and all alloys inside of (or under) the curved surface, outlined by the traces, will show some ferrite separation upon heating near the peritectic temperature. The compositions above (or outside) this limiting surface will remain austenitic to the temperature of incipient melting.

It is interesting to note a peculiar property of peritectic solidification. Compositions which would, when properly homogenized, exhibit no ferrite in the solid state may still contain a small amount when rapidly solidified due to the coring effect. The first crystallites to solidify are so rich in iron (and probably chromium) as to solidify partially as ferrite. With ample time for diffusion these crystallites would react with the melt and transform, prior to complete solidification, to austenite, but such time is not usually permitted, hence ingots may show traces of ferrite in compositions which develop none when the homogeneous worked metal is heated to the peritectic temperature.

An unexpected circumstance, which, at first inspection, seems unlikely, is also observed in these ferrite-forming nickel-chromium alloys. When increasing ferrite is separated by increasing temperature the maximum ferrite proportion is reached just prior to incipient peritectic fusion. Heating even slightly into the solidus-liquidus temperature zone abruptly lessens the amount of ferrite present and recognizable after quenching.

The examinations for high temperature ferrite were made microscopically, in general, although in some cases the amount formed was estimated magnetically by the method described later. The ferrite separated at high temperature has a very characteristic mode of existence in that it coalesces rapidly into pools of similar outline. The attack of the etching reagent (aqua regia-glycerine was used exclusively) is much more rapid upon the ferrite regions than upon the austenite due probably to a lower nickel content; certainly, the two phases must have considerably different compositions. The influence of carbon to restrict the formation of ferrite is more clearly shown as plotted in Figs. 7 and 23. The photomicrographs of Figs. 9, 10 and 11 illustrate the progressive change in the ferrite separation at increasing temperatures. Fig. 11 shows the appearance of the constituent after heating slightly above the peritectic temperature.



Fig. 9—Photomicrograph at 500× Showing Typical Occurrence of Small Proportion of Ferrite (High-Temperature). Fig. 10—Photomicrograph at 500× Showing Typical Occurrence of Abundant Proportion of Ferrite (High-Temperature). Fig. 11—Photomicrograph at 500× of Structure Developed by Heating the Alloy of Fig. 10 above the Peritectic Temperature.

The Allotropic Transformation. B. Low Temperatures

Just as the alloys in the vicinity of the 18 chromium-8 nickel range are not wholly austenitic at elevated temperatures, just so are they not stable as austenite at low temperatures. The characteristic behavior of iron upon cooling to transform from gamma iron to alpha iron is still reflected even in these alloys of iron containing so much of other elements. In the physical-chemical sense, to stabilize the austenite in a low-carbon 18 per cent chromium alloy at room temperature might require probably as much as 25 per cent nickel. But due to the extreme sluggishness of high chromium alloys to undergo any structural change except at quite elevated temperatures

such alloys once rendered austenitic remain so even with the slowest possible cooling. Consequently, when sufficient nickel is added to lower the true transformation point down to a temperature in the vicinity of 600 degrees Cent. (1110 degrees Fahr.) the alloy is, from the practical standpoint of heat treatment, a permanently austenitic material. 8 per cent nickel is quite adequate for this purpose. Carbon also contributes to this retention of austenite. Indeed an alloy containing some 20 or 22 per cent chromium with 0.50 or 0.60 per cent carbon but without nickel heated to form a uniform austenite may be cooled rapidly to preserve a permanent metastable austenite. The sluggishness which permits this very great undercooling is, of course, well known in the case of the high-carbon 12 per cent manganese steels, but it should be borne in mind that it is partly the carbon in this case which permits the undercooling and preservation of austenite; carbon-free high-manganese alloys are partially ferritic even with more manganese present than the Hadfield compositions. Likewise nickel alone does not render its iron alloys wholly austenitic except perhaps some 30 per cent is present. It is quite apparent that the combination of an element, such as nickel or manganese, which lowers the transformation markedly, with an element such as chromium (and carbon) which contributes sluggishness — even though it raises the transformation point—is the effective procedure for developing non-magnetic alloys.

Since the alloys of iron with about 18 per cent chromium and about 8 per cent nickel are not rendered ferritic at any temperature between liquid air and that of austenite stability it may well be asked —are they not then truly stable austenite at ordinary temperature? The answer that they are actually stable only as ferrite (except appreciable carbon be present) follows from two circumstances. The first is only presumptive but serves to cast doubt upon the austenite stability. As nickel is added to an alloy of iron with 12 per cent chromium the transformation point, well above 900 degrees Cent. (1650 degrees Fahr.) is lowered at a certain rate. If now, to such an alloy containing, for example, 3 or 4 per cent nickel wherein the sluggishness is not yet very great, more chromium is added it is found that the critical point is raised slightly. But the effect of nickel to lower the transformation point appears not to be nearly great enough to depress it to room temperature at the 18 and 8 composition. Extrapolation from determinable temperatures into inde-

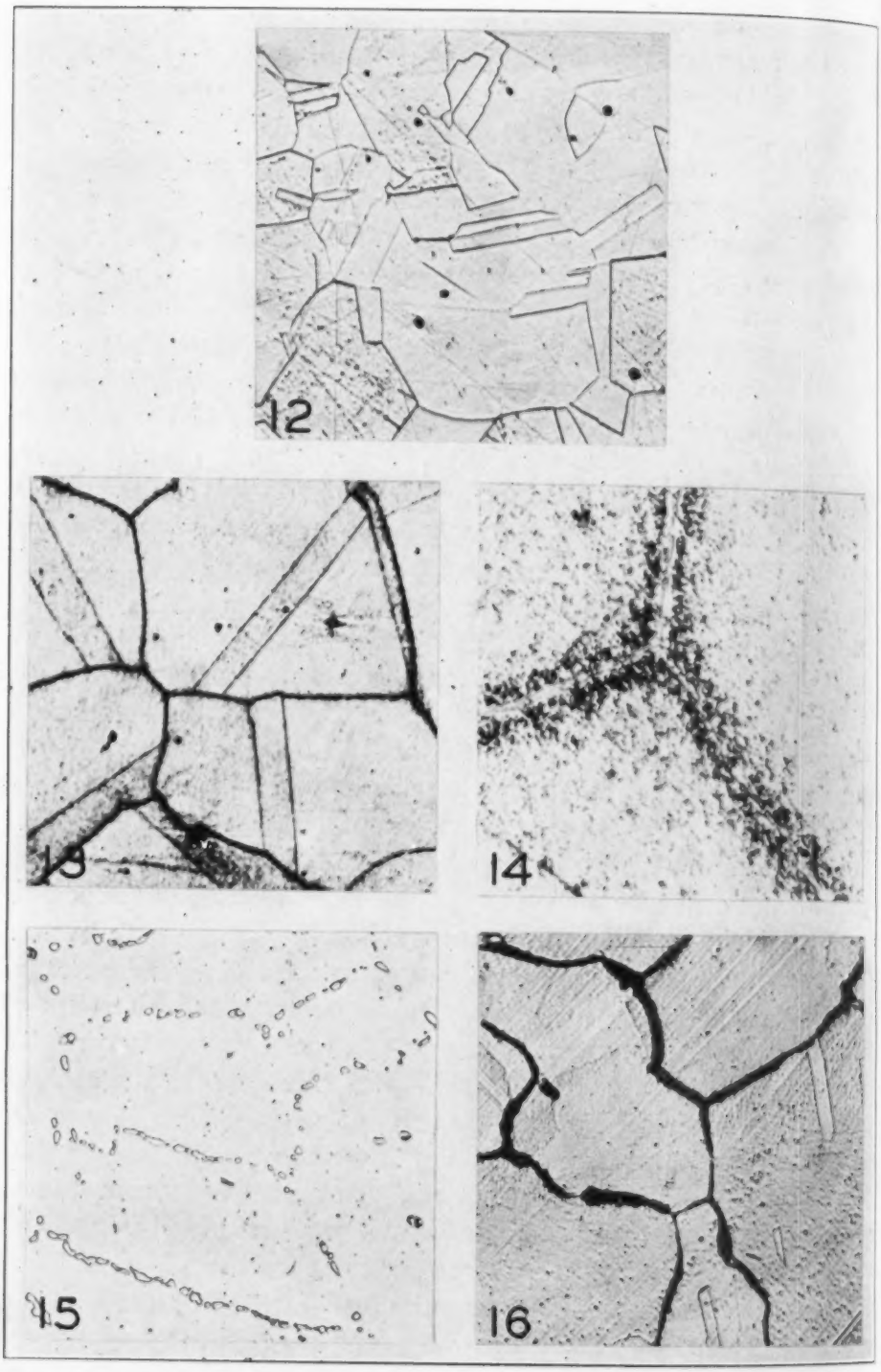


Fig. 12—Photomicrograph at 300× of Normal Structure of the Wholly Austenitic Alloy. Fig. 13—Photomicrograph at 300× Showing Grain Boundary Appearance After Slow Carbide Precipitation at Low Temperature. Annealed Metal. Fig. 14—Photomicrograph at 2500× Showing Mode of Carbide Precipitation at Low Temperature. Annealed Metal. Fig. 15—Photomicrograph at 1500× Showing Coarse Carbide Precipitated at High Temperature. Fig. 6—Photomicrograph at 500 × of Metal Pickled After Low Temperature Carbide Precipitation had Occurred.

terminable ranges is, of course, not valid, but in this case such a sudden change in trend would be required that it seems utterly improbable. The second circumstance is very positive. All alloys of the composition range here discussed may by cold work be rendered partially ferritic and accordingly magnetic. The only effect upon constitution which the agitation by cold work could exert would be to assist in establishment of equilibrium; for it appears that cold work upon plastic crystals exerts essentially the same influence as the stirring of liquids or liquid aggregates. In the case of these iron-nickel-chromium alloys severe cold work may transform a wholly austenitic metal to one which is about 95 per cent ferrite, provided the work is done at low temperatures. The ferrite thus formed is, of course, severely stressed and exhibits the characteristic properties of any cold-worked ferrite. Thus these alloys rendered more or less ferritic by cold work are, so far as allotropy is concerned, in a condition well suited for study of transformation points by heating to successively higher temperatures and observing the decrease in ferrite. The advantage, generally, of this method lies in the slight tendency to superheating in comparison with the general strong tendency to undercooling in all reactions in the solid state.

Strictly speaking, the ideal method for determining the upper temperature limit for pure ferrite formation and the lower limit for pure austenite would be:—

1. Cold work the metal until it becomes essentially pure ferrite; and then 2. Work this metal *at* a series of constant temperatures and estimate the ferrite thereafter. The temperature of first diminution in ferrite would mark the lower limit of the heterogeneous (ferrite plus austenite) field, and the final disappearance of ferrite, the upper limit. In the present investigation a somewhat different procedure was followed. In the first place it was discovered that the effect of cold work to harden the alloy and reduce its ductility is exerted so rapidly that in no case could the alloy be rendered wholly ferritic before it began to develop cold-work cracks. The transformation back to austenite upon heating had in most cases to be estimated upon only partially ferritic material which would tend to obscure the lower limit of the heterogeneous temperature range. Any attempt to remove the excessive cold-work effects without transforming the ferrite developed failed entirely since the lowest annealing temperature is far above the transformation temperature. Furthermore, except in the case of the lowest carbon content materials, the carbide reac-

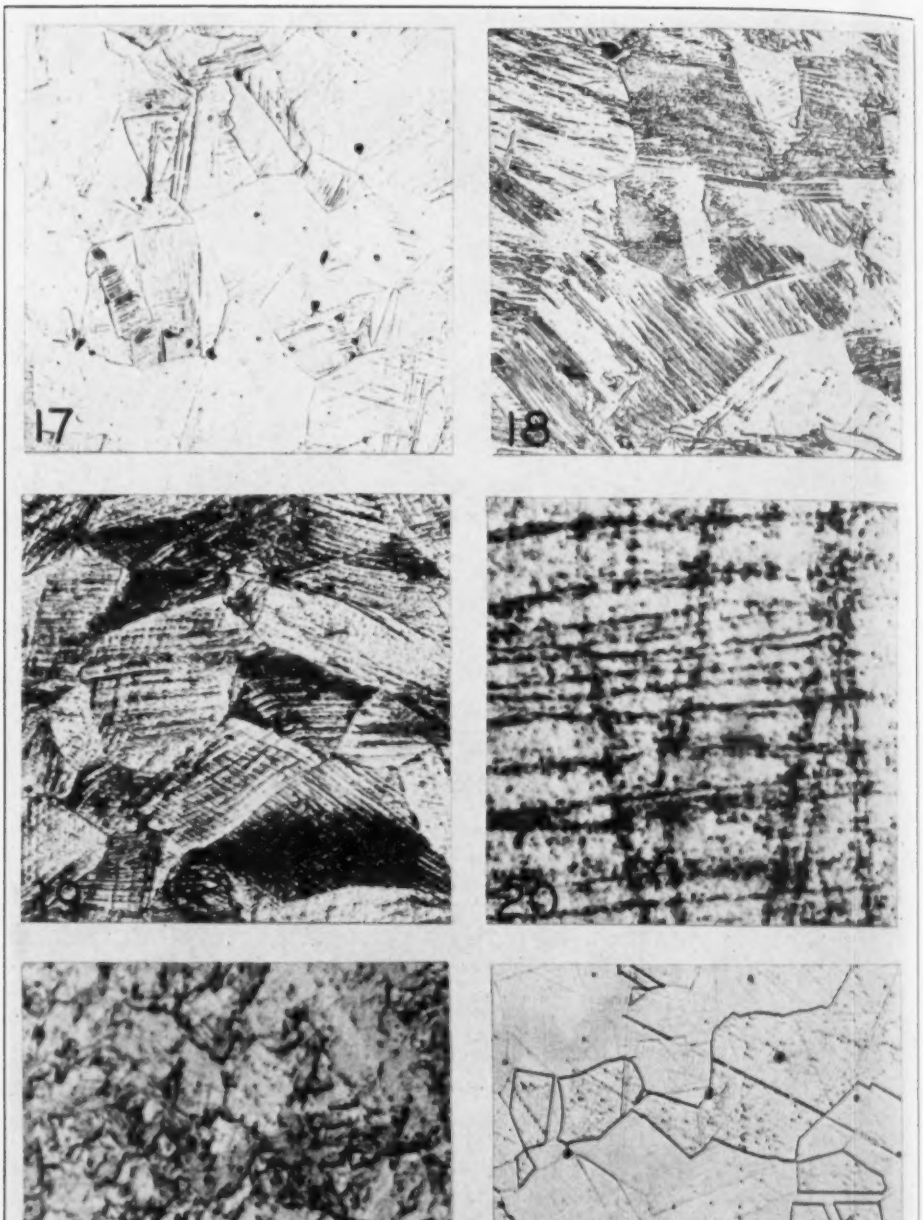


Fig. 17—Photomicrograph at 250× of Cold-worked Stable Austenite. Fig. 18—Photomicrograph at 500× of Cold-worked Unstable Austenite. Fig. 19—Photomicrograph at 500× of Cold-worked Unstable Austenite Reheated to Temperature of Incipient Carbide Precipitation—about 550 degrees Cent. (1025 degrees Fahr.). Fig. 20—Photomicrograph at 2000× of Cold-worked Unstable Austenite Reheated to 625 degrees Cent. (1160 degrees Fahr.). Carbide Particles still Largely Unresolvable. Fig. 21—Photomicrograph at 2000× Showing Incipient Recrystallization After Cold Work and Carbide Precipitation—about 825 degrees Cent. (1520 degrees Fahr.). Fig. 22—Photomicrograph at 300× Showing Completely Recrystallized Structure. Carbon in Solution.

tion, to be discussed later, set in and vitiated the results. Furthermore it was found almost entirely impracticable to work the metal at any well controlled constant temperature in the small specimens used. But maintaining the previously worked material at a constant temperature without the aid of any work was found to suffice to bring about equilibrium if the time was sufficiently long. The results presented here then represent the temperature zone wherein

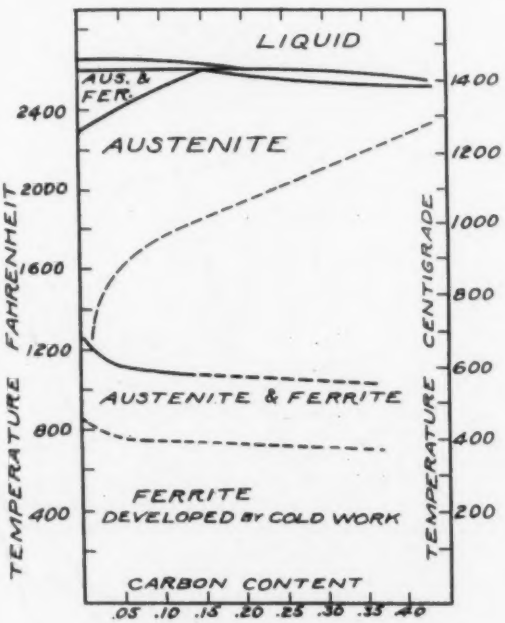


Fig. 23—Chart Showing Approximate Austenite-Ferrite Transformation Temperatures in Equilibrium Condition.

the ferrite formed by cold work gradually disappeared as the temperature was raised in steps—the time at temperature being sufficient to bring about equilibrium.

The estimation of ferrite formed through the agency of cold work is not possible by microscopic examination. There is a slight though characteristic difference between the appearance of the cold-worked structure of unstable austenitic metal and the appearance of cold-worked stable austenite. The ferrite formed upon the slip planes is actually manifest by a characteristic appearance at high magnification; but a quantitative estimation would not be possible. A comparison of Fig. 17 with Fig. 18 may suffice to show this difference. So far as is known only two methods are really applicable, X-ray analysis and magnetic permeability measurements. The X-ray

measurements are, of course, made upon the relative intensity of the ferrite and the austenite patterns which is an absolute function of their respective amounts. But the quantitative estimation of intensities is one which holds possibility of some considerable error. The magnetic permeability determination is a more nearly objective measurement and capable of high precision but here the absolute

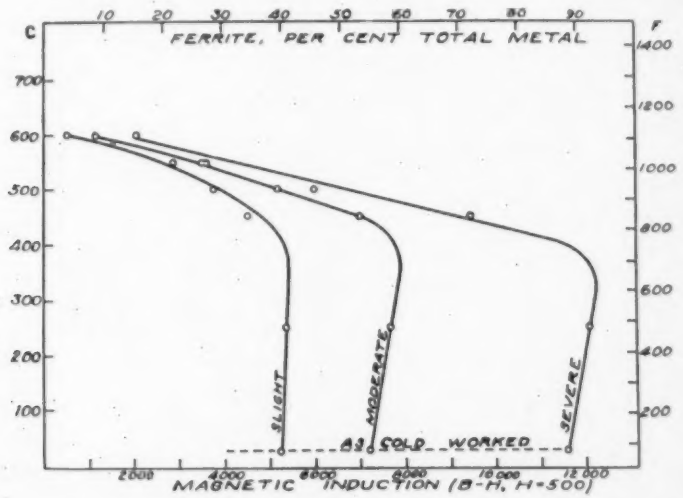


Fig. 24—The Restoration of the Austenitic Condition by Heating After Cold Work. Magnetic Induction and Ferrite Proportion at Equilibrium Plotted against Heating Temperature.

correlation between amount of ferrite and permeability cannot easily be made.

To secure the advantage of both methods about 45 specimens whose ferrite content ranged (by virtue of composition and heat treatment) from a trace to about 95 per cent were studied by both methods. A correlation by averages could then be set up. It is therefore estimated that pure cold-work ferrite of 18 and 8 composition would show a net magnetic induction of about 13,000, H = 500. From these correlations it was found that magnetic permeability and proportion of ferrite as determined by X-ray examination are substantially, though not exactly, in linear relation.

The result of the investigation of the ferrite-austenite heterogeneous temperature range for a typical alloy is shown in Fig. 24. In this case three different degrees of cold work were applied to the metal and the effect of reheating observed. As may be seen, the amount of ferrite is actually increased by heating up to about 400 degrees Cent. (750 degrees Fahr.); this temperature then is taken

as the lower transformation limit for this particular composition. The increase is thought to be due to the production of a slight amount of ferrite by the release of stresses not maintainable at the higher temperature at which the metal has lower strength and hardness. This slight increase in ferrite accompanied by hardness increase is brought about very quickly, while the diminution in ferrite content accompanying higher temperatures is a slower process. The time interval required for the establishment of equilibrium at the higher

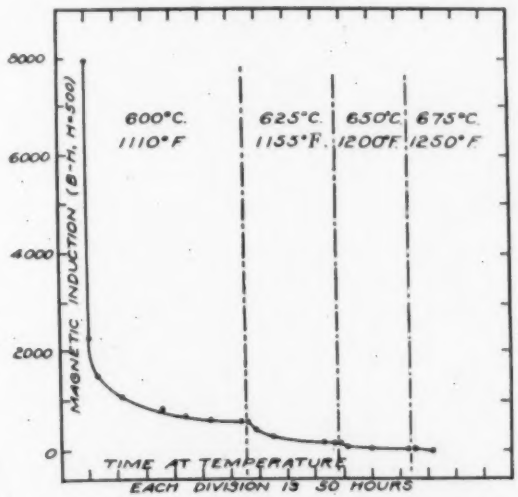


Fig. 25—Time Required for Establishment of Equilibrium at Various Temperatures of Heating as Indicated by Magnetic Induction. Alloy Contains about .02 per cent Carbon.

temperatures within the temperature zone of mixed phases may be observed in Figs. 25 and 26, in which the change in permeability with time at successively applied temperatures is plotted. It is clear that equilibrium was essentially established at several temperatures. Fig. 25 represents a very low carbon alloy, while Fig. 26 represents the elimination of ferrite in an 0.08 per cent carbon alloy and illustrates the effect of carbide precipitation.

In the investigations several field strengths were used for each determination but only one is plotted. The reason for employing several field strengths was to establish something of the nature of the virgin magnetic curve which varies with the condition of the ferrite in a characteristic manner. In general the evidence was indicative of a somewhat martensitic condition in the lower temperatures and a more nearly annealed ferritic condition in the magnetic material remaining at the somewhat higher temperatures. The relative

induction at high and low fields offers this differentiation. This and other details of the ferrite-austenite transformation were checked by some 200 X-ray crystallograms.

Except in the case of the lowest carbon content alloys the precise temperature of ferrite disappearance could not be determined. At temperatures very near that required for complete restoration of the austenite, carbide usually began to precipitate, and while this be-

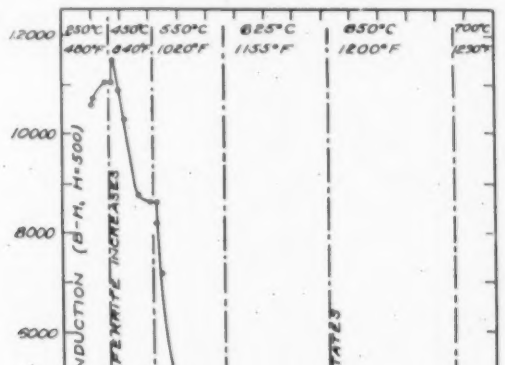


Fig. 26—Elimination of Ferrite by Heating After Two Degrees of Cold Work. Equilibrium Reached for Each Temperature. Note Effect of Carbide Precipitation. Alloy Containing .08 per cent Carbon.

havior alone would not necessarily be serious, in many cases the carbide precipitation is accompanied by the formation of a small amount of abnormal ferrite which is not properly regarded as a part of the allotropic behavior of the alloy. As will be discussed later this ferrite is depleted of its carbon content and probably also impoverished in its alloy content so that it is not a phase of composition represented by the analysis of the alloy. When carbide precipitation caused abnormal ferrite precipitation to occur simultaneously in these magnetic and X-ray specimens the particular investigation was discontinued and an extrapolation to zero ferrite was made on the curve.

Accordingly the exploration is not free from a possible error in some cases, although usually the ferrite could be eliminated in such a short time that abnormal ferrite in significant amount had not yet developed. Higher nickel content almost entirely prevented the appearance of abnormal ferrite even though carbide precipitation did occur. Chromium content over the range studied had little or no effect upon this abnormal ferrite development.

Among the details of behavior involved in the ferrite-austenite transformation there is one of significance sufficient to warrant its mention. There is evidence that in these low-temperature reactions between the two crystalline phases, the ordinary differences in composition between the two either do not exist at all or are essentially nullified by the conditions. To explain, if the entire mass of the alloy under investigation were in true equilibrium, and if diffusion were able to occur with facility, then there would be a substantial difference in composition between the two phases, ferrite and austenite. Accordingly, if a small amount of ferrite were developed its proportion would not vary with heating until such temperature was reached as would correspond with the particular amount of ferrite present. Such, however, is not the case here as shown in Fig. 24, for each specimen with its particular ferrite concentration traces its own complete temperature-constitution curve. The only conclusion which may be drawn is that the unchanged austenite plays no part whatever in the reaction and only the ferrite developed by cold work enters into the reversed transformation. In a sense, then, each curve of Fig. 24 is precisely a copy of the other with a different amount of ferrite functioning just as though it were 100 per cent. This circumstance throws considerable light upon the whole mechanism of the low temperature transformation. It is evidently carried out without more than traces of diffusion below about 500 or 550 degrees Cent. (930 or 1020 degrees Fahr.).

With this observation in mind the microscopic appearance of the cold-worked alloys takes on added significance. The cold-worked structure is not altered by heating short of about 400 degrees Cent. (750 degrees Fahr.). Heating above this temperature seems to develop a great many, just resolvable parallel fragments of a different constituent in the slip planes—presumably ferrite. The proportion of this constituent, if such it may be termed, increases with the degree of cold work or with the magnetic permeability. It is too vague to photograph well. Heating to somewhat higher temperature does

not change this appearance at all though the ferrite is gradually disappearing. The appearance remains essentially the same except the alloy develops carbide separation or ultimately recrystallizes. The inference is that diffusion is so difficult that these tiny platelets of ferrite simply transform to austenite upon heating above 400 degrees Cent. (750 degrees Fahr.) *in situ* and are unable to merge with the surrounding austenite except at much higher temperatures. The microscopic evidence and the magnetic determinations of the progress of the ferrite—austenite transformation being in such perfect agreement may perhaps warrant the introduction of this description of a rather unique metallographic behavior although admittedly it is of exclusively scientific interest.

The carbon influence upon the low-temperature ferrite is quite simple. Increasing carbon diminishes the amount of ferrite produced by a definite degree of cold work. But the amount of cold work possible is not noticeably greater with higher carbon content since the material hardens about as rapidly. The presence of higher nickel reduces the amount of ferrite formed and the alloy does not harden so rapidly with cold work. These circumstances may be explained on the basis of the formation by cold work of a ferrite supersaturated with carbon in the same condition as in martensite. The higher carbon content of the ferrite formed in higher carbon alloys would, of course, make a harder product and more rapidly reduce the plasticity with cold work. Higher nickel content, by reducing the amount of ferrite transforming, would lessen the degree of extraordinary hardening. The particular alloy whose behavior was represented in Fig. 24 developed a hardness by cold working of about 50 Rockwell C accompanying the production of about 90 to 95 per cent ferrite. This particular specimen showed considerable permanent magnetism after the severest cold work followed by magnetization. Obviously this degree of hardness would not be produced by any ordinary deformation-hardening process.

The low carbon alloys when heated showed a less rapid loss of ferrite with temperature than the higher carbon alloys. The nickel effect has already been shown for alloys of negligible carbon in Figs. 5 and 6. There remains then to depict the probable relation of carbon content to the allotropic transformation temperatures and this is shown in Fig. 23. The lines bounding the heterogeneous field are probably correct for the very low carbon alloys but are not nearly so reliable as the carbon content increases for the reasons mentioned.

However, it is certain that the temperatures of the transformation zone decrease with increasing carbon to a greater or less degree.

A word of caution in reference to the interpretation of Figs. 7 and 23 is perhaps in order. These charts are in a sense plane sections of a four-dimensional system and, while entirely consistent in representing a single behavior as evidenced in the compositions shown they are not binary equilibrium diagrams and cannot be used as such since, for example, the precipitation of any carbide whatever or the transformation of any of the austenite introduces compositions not existsenting a single behavior as evidenced in the compositions shown, they serve satisfactorily for portraying the behaviors. For convenience the carbon solubility line is traced on Fig. 23.

Carbon Influence Upon Corrosion Attack

Since the inauguration of the work described here an interesting article[6] has appeared on the change in properties of these stainless alloys brought about by carbide precipitation. In this paper Strauss and his co-workers describe very clearly some of the manifestations of the reprecipitation of the carbide dissolved at elevated temperatures. In the main the observations which the present authors had gathered are fully corroborated by their evidence, although in some instances somewhat different explanations have been offered. In view of the thoroughness of the description of the phenomena of carbide precipitation in that paper the present description is made as brief as possible.

Carbide in Annealed Metal

The solubility curve of the carbide in the nickel-chromium alloys at once indicates that it is an example of a precipitation hardening alloy, falling in the general class of duralumin, Tempaloy, and Sykes' iron-tungsten and iron-molybdenum alloys. In all these materials the alloys are first heated to a high temperature to dissolve completely the element or elements capable of forming separate crystallites upon cooling. The solid solution formed at high temperature is quenched to retain the pure solid solution in a state of supersaturation. Subsequently the metal is reheated to a temperature sufficiently high to permit diffusion but not high enough either to restore the stable solid

[6] Loc. cit. (reference 4).

solution nor to coalesce the particles into large and hence ineffective masses.

In the case of the chrome-nickel rustless alloys the solution and precipitation occurs as in the hardenable alloys and indeed the hardness is actually raised by suitable quenching and reheating to a degree depending upon carbon content. There is one outstanding distinction between the behavior of these alloys and the others which are employed for their hardenability. In the typical hardenable alloy the precipitation is quite general throughout the grains but carbide precipitation in the rustless chromium-nickel steels occurs predominantly in the grain boundaries. As a result when precipitation occurs, the loss in ductility is more marked than the gain in hardness. The more general the precipitation of the hardenable alloys the greater the trend toward maximum hardening by precipitation with minimum loss in ductility. However, the worst precipitation of carbon in most of the "18-8" compositions reduces the impact and elongation only to perhaps half of its original value as a general rule.

The results of carbide precipitation are observed in the mechanical properties, the magnetic properties and in the reaction of the metal in corrosion and oxidation attack. The mechanical properties are somewhat altered as briefly stated above. The magnetic properties, depending upon the existence of ferrite, may or may not be altered, depending upon the temperature at which the precipitation of carbide occurs and upon the composition of the alloy. These effects are of relatively less importance than the change in chemical properties of the alloy.

Fig. 13 shows at moderate magnification, and Fig. 14 at high magnification, the microscopic appearance of the grain boundaries wherein the carbide has clearly precipitated at relatively low temperature. Fig. 15 shows the more perfectly coalesced carbide as precipitated from a higher carbon alloy as a result of treatment during which the carbide was chiefly rejected at rather high temperature. As will be seen the former is a more disadvantageous condition than the latter.

A surprising mode of attack follows when metal in which the carbide has precipitated at low temperature in voluminous amount is subjected to an acid solution. The photomicrograph of Fig. 16 illustrates the structure of such abnormal metal subjected to pickling acid. It is at once evident that the attack has taken place almost entirely

in the grain boundary regions, or, in short, just where the carbide precipitation occurred. Indeed prolonged acid attack may remove all intergranular metal so that the individual grains fall apart as a powder. In the case of metal of similar high carbon content but in which the carbon is in solution the attack would be normal—the grain boundaries only moderately engraved below the average surface. Almost any sort of acid corrosion will proceed to develop the exaggerated inter-granular attack, but the effect is much more pronounced with certain reagents. If there is some moderate oxidizing agent present the effect is more pronounced, apparently due to the protection developed upon the grains which as a whole are more or less normal.

In attempting to develop a theory to account for this rather unique grain boundary attack there are two factors which must be borne in mind. First, carbide precipitation may be brought about at temperatures fully as low as 600 degrees Cent. (1110 degrees Fahr.) in an alloy whose recrystallizing temperature (after cold work) is at least 900 degrees Cent. (1650 degrees Fahr.). There is therefore very little general diffusion of the principal alloying elements at the temperature of carbide precipitation. It is quite clear on the other hand that carbon may migrate at a somewhat more rapid rate. There must then be a very considerable difference in the diffusion rates of chromium and carbon. The other prominent factor is the composition of the carbide precipitated. So far as is known no determinations of the composition of the carbide separating out of the austenitic stainless alloys have been made. Indeed, were it not that the precipitation parallels in quantity the carbon content of the alloy there would be little reason to assume that the precipitate is a carbide. But since it seemingly is a carbide the composition must then in all probability be Cr_4C or more properly $(Cr + Fe + Ni)_4C$, for Westgren[7] and others have found that in high-chromium iron alloys the carbide to separate is the crystalline entity Cr_4C with some iron replacing chromium. It is unlikely that the presence of some 10 per cent nickel would change anything except the actual composition of this crystal by substituting some nickel as well as iron in the structure of the carbide crystal.

The chromium content of this precipitated carbide would then be very high—certainly in the vicinity of 90 per cent. A partial

[7] A. Westgren, G. Phragmén och Jr. Negresco. Om Järn-krom-Kollegeringarnas Byggnad. *Jernkontorets Annaler för Ar* 1927.

electrolytic separation of such carbide which was necessarily not free from solid solution showed a chromium content of about 75 per cent. Each per cent of carbon present may then, upon precipitation, remove from 15 to 19 per cent chromium from the solid solution. But, as has been postulated, chromium may not migrate freely in these alloys while carbon obviously does do so as evidenced by the voluminous grain boundary separation. Hence, the conclusion is more or less imperative that as soon as carbide precipitates in the grain boundaries the immediately adjacent metal must be very greatly depleted in chromium. Without chromium to develop its unique inert protective surface the depleted metal would offer far less resistance to almost any sort of corrosive or oxidizing attack. The local impoverishment of grain boundary metal in chromium is therefore suggested as the cause of the inordinate grain-boundary attack when voluminous carbide has been precipitated.

If the carbon in excess of the solubility limit is precipitated at a higher temperature at which, in the time permitted, chromium may diffuse, then the grain boundary metal can be replenished from the large chromium supply within the grain and the inordinate attack should not occur in the grain boundaries. If the carbon is in the lower range, for example, around 0.08 per cent, the total amount of the chromium removed is in fact not great and with the customary analyses would still be sufficient *if evenly distributed* to offer its protection against corrosion attack. For example, if the alloy is heated at say 875 degrees Cent. (1600 degrees Fahr.) for a considerable time the carbide in excess of say 0.06 per cent will precipitate, but subsequently the regions thus depleted of chromium can be restored if time is allowed. Subsequent heating at a lower temperature then will find very little further carbon with which to form carbide and any further grain boundary alteration will be slight and extremely slow. Some actual tests have demonstrated the general validity of this prediction and this in turn serves to confirm the correctness of the picture of the mechanism of grain boundary corrosion. The practical value of this "desensitizing" treatment is not yet fully determined.

It will be obvious at once that the intergranular attack by corroding media may be eliminated either by preventing carbide precipitation (e. g., lowering the carbon content or somehow raising its solubility) or by precipitating it permanently under conditions which will not remove chromium from solid solution so drastically at any points.

Carbide in Cold-Worked Metal

In the immediately preceding paragraphs the procedure of carbide precipitation has been described for the annealed metal free from cold-work effects. Carbide precipitation does however occur, even more rapidly, upon reheating to temperatures above about 550 degrees Cent. (1020 degrees Fahr.) the austenitic alloys which have previously been cold-worked. The point has been stressed that carbide precipitation in the alloys studied occurs predominantly almost exclusively in the grain boundary regions. As may be seen in the photomicrographs even the planes of junction between the twinned zones of the individual grains are not able to attract much carbide. The same, however, may not be said for slip planes. Carbide is precipitated very readily upon reheating on all the slip bands produced in cold working provided sufficient carbon is present. This fact has already been mentioned in connection with the studies of the transformation points of the alloy. Depending upon the composition of the alloy the carbide precipitation is more or less definitely indicated in the etching of the metal properly polished for metallographic examination. Without any magnification the carbide precipitation is usually recognizable by the dark surface of the normally etched polished surface. If one observes the time required to etch the specimen to a certain average brilliance under the microscope the influence of carbide precipitation is most pronounced. A reasonably well smoothed surface of the metal is also amenable to the use of another sort of indicator. When submerged in various corroding reagents such as $FeCl_3$ — HCl solution, H_2SO_4 or $H_2SO_4 + CuSO_4$ solution, etc., the cold-worked specimens free from carbide precipitation remain bright even if attacked slightly, while those same compositions which have been reheated to sufficiently high temperatures long enough to cause precipitation will become coated with a dark gray-brown or black sludge depending upon the composition and volume of carbide precipitation. The authors preferred the use of $FeCl_3$—HCl solution. Usually, after many observations and comparisons of the microscopic appearance, this method becomes the best criterion of carbide separation but is not in any sense absolute without control specimens. The photomicrographs of Figs. 19, 20 and 21 show the appearance of cold-worked metal in which carbide has precipitated.

The precipitation of carbide here occurs at least 300 degrees Cent. below the so-called recrystallizing temperature and, just as in

the annealed metal, diffusion of the chromium is very slow. But in this case the conditions are quite different. The slip bands which in their moment of formation may become badly crystallized ferrite are quite close together even when the metal is only moderately deformed. Presumably, carbon precipitates first from the supersaturated ferrite since it is generally supposed that the crystal form of ferrite holds less carbon than does austenite. At any rate, when the metal is severely worked and networks of carbide precipitation appear, chromium would have to diffuse through an extremely minute distance—at the most a matter of thousandths of a millimeter—to replace that removed as carbide. Even at low temperatures then the deep etching attack might conceivably be eliminated by a heating to restore partial homogeneity.

It has been observed that cold-worked specimens regain their bright etching character as well as other behaviors indicating restored (or partially restored surface passivity) after long heating at temperatures below that for the complete solubility of carbide. The foregoing speculation would explain how the re-establishment of substantial chromium homogeneity could account for this behavior. It appears that the first trace of carbide precipitation—when the particles are sub-microscopic in size—always causes some change in the delicate balance between the rate of protective surface formation and the rate of the opposing tendency for the metal atoms to leave the metal by corrosion. It is ultimately the domination of one or the other of these two tendencies which determines in how far a material is resistant in any certain environment. While carbide precipitation is almost always manifest by some change in the electro-chemical behavior of these alloys it is not always accompanied by any development of magnetic properties. This would tend to indicate that it is not the development of ferrite *per se* which influences the corrosion behavior.

To stress this point further it is only necessary to point to the chemical inertness of certain of the alloys of high chromium content which exist normally as a microscopic aggregate of ferrite and austenite. But in addition there is also the fact that the 18 and 8 composition when cold-worked to develop more ferrite than austenite has still excellent corrosion resistance and is not darkened by any of the reagents described above. In view of these circumstances the belief just outlined may be justified although it differs from the statement of Strauss ". . . . the more probable explanation of the intercrystalline

corrosion will not be the carbide formation but presence of alpha iron."

The present authors therefore have taken the evidence to indicate that carbon precipitation occurs by relatively rapid carbon migration to the regions of atomic freedom, i. e., grain boundaries and slip planes. The formation of the carbide requires such a high proportion of chromium that the carbide is formed by impoverishing the

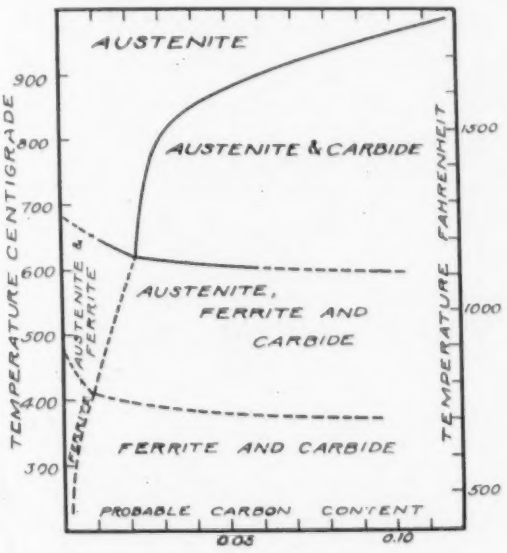


Fig. 27—Portion of the "18-8" Section of the Iron-Chromium-Nickel-Carbon System Showing Probable Phases at Equilibrium as predicted from Observations.

immediately adjacent metal with respect to chromium (and perhaps nickel to a slight extent) to some concentration below that adequate for surface-layer protection. This permits of potential grain boundary attack or even of general attack depending upon the state of the metal—annealed or cold-worked. Subsequent heating may, in cold-worked metal, restore chromium by diffusion even with carbide still precipitated, and to a lesser extent, even in annealed metal wherein the path of chromium diffusion is longer. The corrosion effect is largely independent of any ferrite development, except that when ferrite is formed the depletion is probably more severe and restoration more difficult. When the nickel content is relatively high (see Fig. 5) or the carbide precipitation temperature rather elevated, then no ferrite accompanies carbide separation.

An interesting metallographic behavior enters into the mechan-

ism of carbide precipitation. It is frequently observed that ferrite is produced as an accompaniment to carbide precipitation at temperatures as high as 750 to 800 degrees Cent. (1380 to 1470 degrees Fahr.). According to the curves of Fig. 23 no ferrite may exist as temperatures above about 675 degrees Cent. (1250 degrees Fahr.) even with low carbon content. This apparent discrepancy hangs again upon the distinction between stable and metastable equilibrium. In a four-dimensional equilibrium diagram the carbide phase would be shown present in equilibrium with ferrite and austenite but the ferrite and austenite would be of very different compositions. When now the extreme metastability due to slow atomic migration of the system is considered the entire situation becomes hopelessly complicated. The most promising way to treat the subject seems to be to introduce a section of the probable equilibrium diagram and to describe the sequence of events for a single alloy. The section of the equilibrium diagram is shown in Fig. 27. When a specimen of the austenite alloy with, for example, a composition of 18 per cent chromium, 8 per cent nickel and 0.08 per cent carbon is heated slowly from room temperature after some ferrite has been developed by cold work the various phenomena occur in the following manner.

1. To about 400 degrees Cent. (750 degrees Fahr.). Incipient coalescence of ferrite and slight increase in ferrite volume.

2. From 400 to 600 degrees Cent. (750 to 1110 degrees Fahr.). Gradual transformation of ferrite *in situ*. Newly formed austenite does not merge with residual austenite. Only a little ferrite remains. In general incomplete adjustment of composition takes place.

3. From 600 to 800 degrees Cent. (1110 to 1470 degrees Fahr.). Carbide precipitation occurs and development of ferrite accompanies the action. Ferrite may be nearly pure iron since the chromium is removed by the carbide and the nickel is partially rejected by its greater solubility in austenite. In this range a very long time at constant temperature will cause disappearance of ferrite. Carbides gradually coalesce to sizeable particles at highest temperature of range. Constitution is austenite plus carbide.

4. From 800 degrees Cent. (1470 degrees Fahr.) to about 980 degrees Cent. (1800 degrees Fahr.). Carbide gradually disappears and recrystallization occurs.

5. From 980 to 1275 degrees Cent. (1800 to 2325 degrees Fahr.) the metal undergoes no change except grain growth.

6. From 1275 degrees Cent. (2325 degrees Fahr.) to peri-

tectic melting point the metal gradually expels ferrite of composition fairly remote from the main portion of alloy. Maximum ferrite just below incipient fusion may be 15 to 20 per cent.

Significance of Carbide Precipitation

The evidence seems to point to a possibility of carbide precipitation even when the carbon content is well under 0.04 per cent. To discover this minute separation of carbide requires the closest scrutiny under the microscope, for its effect upon ordinary corrosion behavior is too slight to be observed. Accordingly it is clear that

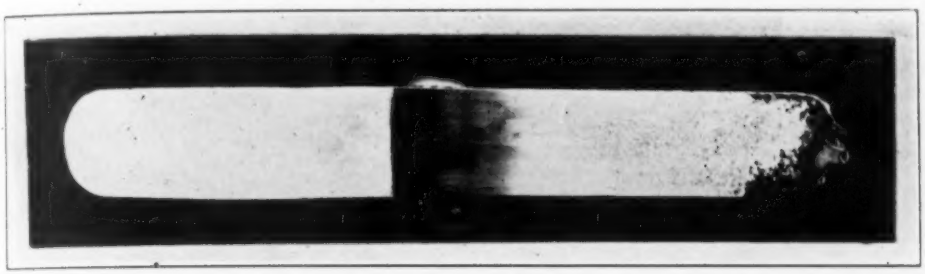


Fig. 28—Typical Corrosion Behavior of "18-8" Strip in Three Conditions: Left, Cold-worked; Right, Annealed; Center, Low-Temperature Carbide Precipitation. Temperature Gradient Specimen.

the mere fact of carbide precipitation does not always indicate any significant reduction in practical corrosion resistance. And even when the carbon content is sufficiently high so as to constitute a potential source of the intergranular corrosion the actual condition is not manifest except the precipitation is brought about by long heating in the particular temperature range required.

Consequently, no definite general statements are properly made concerning the deleterious effects of composition and handling. Instead, it is necessary to consider separately each application as it falls into a definite class of conditions in service or fabrication. It may be safely stated that over a considerable range carbon content is of little importance except the metal is to be heated in the temperature range of fine carbide precipitation. The long time exposures to the lower temperatures of the range are apparently the most serious.

Cold work in itself seems to have little effect upon the corrosion resistance in the cases where this is very high. As with all other metals, the cold-worked metal dissolves in reagents which act vigorously somewhat more rapidly than the annealed metal. An

interesting example of the chemical attack upon the three conditions: A, cold-worked state, B, carbide-precipitation state, and C, annealed state, may be seen in a single specimen as in Fig. 28. The bar shown here was an "18-8" composition with about 0.08 per cent carbon. It was severely cold-worked and then with one end held in cold water the other end was heated to the fusion point and so maintained for about 12 minutes. The whole specimen was then polished and immersed in the $FeCl_3$ — HCl solution for accelerated attack. The left hand end is, therefore, as cold-worked, the middle heated in the range for carbide precipitation and the right hand end above the temperature range of carbon insolubility. All possible intermediate temperatures are represented. The abrupt edge of the blackened zone on the left and the gradual recovery of the bright appearance on the right are self explanatory. Neither the cold-worked nor the annealed structure is attacked noticeably. The blackened center zone, however, is in a condition which would not offer proper corrosion resistance in many environments.

For severe cold-work applications a higher nickel content would often be more applicable since it hardens less rapidly, but if high strength must be developed it is not particularly beneficial. Oxidation resistance at elevated temperature increases with increase in chromium content as in the case of alloys not containing nickel.

A Few Details of Experimentation

The alloys in the vicinity of the 18 and 8 compositions possess a few properties which are almost unique and may at first offer obstacles to the experimentation by others as they did to the present studies.

1. The preparation of the specimens for microscopic examination involves some sort of cutting and the effect of this machining as well as the effect of the coarse polishing media is surprisingly great. So much of the surface layers of the metal is transformed that usually an annealed specimen shows strong markings resembling cold work even when great care is exercised. Deep etching followed by polishing several times repeated is the only method discovered to obtain unmarked, polyhedral grains in the annealed metal. The lower the alloy content (particularly nickel) the greater this effect. It has been observed that those grains whose octahedral planes lie in the plane of the section suffer most in this respect.

2. In making magnetic measurements the "as-machined" samples show a magnetic value too high to be representative of the metal. Ferrite is formed in the surface layers even by fine emery paper. The surface should be treated by immersion in some reagent which etches as uniformly as possible to remove a few thousandths of an inch of the surface metal.

3. The oxide which forms on heat-treated specimens is also quite magnetic. This must be removed. Preferably mechanical methods should not be used, but in any case, the specimens for magnetic examination must be treated in a pickling solution—preferably containing an effective inhibitor.

Acknowledgments

The authors are indebted to several members of the staff of the United States Steel Corporation Research Laboratory, in particular to Mr. W. S. N. Waring for many magnetic measurements; and also to the Illinois Steel Company and the American Sheet and Tin Plate Company, both for many excellent samples of commercial metal and for specially prepared compositions as well as other assistance. A large proportion of the X-ray study on the alloys was performed in the metallurgical laboratory of Harvard University and the authors are grateful to Professor Albert Sauveur for this valued courtesy.

DISCUSSION

Written Discussion: By Robert H. Aborn, Research Laboratories U. S. Steel Corp., Kearny, N. J.

Drs. Krivobok and Grossmann are to be congratulated for another excellent piece of work supplementing their comprehensive investigations of the iron-chromium-carbon system, extended this time to the effects of nickel in the composition ranges now commanding considerable attention. Possibly only those working in this field can realize the extent of the work necessary to adequately investigate such a range of alloys.

It is difficult for one accustomed to carbon steel behaviors to fully appreciate the inordinate sluggishness of these alloys, particularly as the nickel and chromium content approach some 10 and 20 per cent, respectively. Nor does this sluggishness seem to be confined entirely to temperatures below 1600 degrees Fahr. It has been our experience that at least *several* hours of heating are required to reach an approximate equilibrium condition at temperatures below 2000 degrees Fahr., if the carbon content is 0.2 per cent or higher. This is particularly true if the mode of occurrence of the carbon prior to heating is in the form of coarse precipitated carbides. Now, in view of the character of the preliminary treatment (several hours at

1800 degrees Fahr.) given the authors' specimens it would seem quite possible that there might have been some coarse carbides produced in the higher carbon ranges which could not be brought to approximate equilibrium in the subsequent 1—1½ hour treatments. It would seem that this circumstance may serve to explain the few apparent anomalies indicated in Figs. 4, 5, and 6 in which carbides seemed to be present above the probable course of the carbide solubility curve. Furthermore, due to this same extremely low atomic mobility it seems probable that with longer heating times the course of the hardness curves in Figs. 1B and 1C might be somewhat altered, reaching and maintaining a substantially flat minimum above a quenching temperature of about 1950 degrees Fahr.

In connection with the unique set of magnetic induction curves in Figs. 28-31 it would be interesting to learn more fully the authors' interpretation of the observed behaviors. One point is especially worthy of note in these curves, and that is the appearance of high-temperature (delta) ferrite, which is strikingly evident in the sudden upward trend of the curves in Fig. 31, where the carbon content is low enough to cause the appearance of this phase.

In view of the considerable time required for recrystallization as well as carbon solution in these series of alloys it would be interesting to know if the authors have observed any shifting of the magnetic induction curves toward lower values through longer time than the indicated half hour period at temperatures between 1800 degrees Fahr. and 2200 degrees Fahr.

Finally, it may be mentioned that the apparently anomalous magnetic behavior of Alloy 804 (Fig. 31) would seem to be explained through the unusual course of the ferrite-austenite transformation line (counterpart of A_3) in very low carbon content 18-8 alloys as is described in the Bain-Aborn paper on "The Nature of the Nickel-Chromium Rustless Steels" published in the current volume of the A. S. S. T. TRANSACTIONS.

Written Discussion: By J. J. Kanter, metallurgical department, Crane Co., Chicago.

In reading the preprint of the paper "Influence of Nickel on the Chromium-Iron-Carbon Constitutional Diagram" by Drs. Krivobok and Grossmann, I was particularly interested in the results of the magnetic tests which are reported for the 18-8 alloys with various carbon contents. According to the theory of Strauss, Schottky and Hinnuber, referred to in the text, I take it that an alloy with 0.05 per cent carbon content should show less carbide precipitation upon heating at 1200 degrees Fahr. than material with higher carbon. The data shown in Figs. 30 and 31 of the preprint do not seem entirely in accord with the theory if magnetic flux density is an index to carbide precipitation. Alloy No. 804 shows consistently higher flux density after 250 hours heating at 1200 degrees Fahr., for all the previous quenching treatments, than does alloy No. 814. Does this mean that alloy No. 804 is less stable than No. 814? I would appreciate very much further discussion on your interpretation of this apparent anomaly.

Written Discussion: Norman B. Pilling, International Nickel Co., Bayonne, N. J.

This is a very useful contribution to the metallography of the 18 per cent chromium, 8 per cent nickel type of stainless alloy which is now so conspicuously before the public eye. Those interested either in the use or the production of the material will do well to ponder carefully the opinions of the authors, which are evidently the result of much thoughtful consideration.

Interesting and important as the scientific facts are, it does not seem unreasonable to emphasize a few points of practical interest which may be gleaned from this discussion. The extreme versatility of the chrome-nickel alloys of this type is a liability as well as an asset, as it leads to a diversity of applications making it difficult to embrace within a single composition every quality which these uses require. On the other hand the interests of the consuming public would perhaps be served by avoiding too great a variety of grades. It is probably true that the tonnage demand at present is for a material having a pretty appearance, that is, a persistently shiny surface coupled with properties which let it be worked without trouble. The ferritic precipitations which Bain and Aborn describe in the 8 per cent nickel content alloy are unfortunately ores which directly impair the utility of this alloy, that is, interfere with the ease and success with which it can be worked both hot and cold. The authors correctly infer that a rise in nickel content rectifies this difficulty, and it might be added that in spite of the theoretical uncertainties concerning the matter, this is accomplished for all practical purposes by placing this nickel content at about 12 per cent. The present trend of development of the stainless alloys is in the direction of the more stable, slightly higher nickel contents.

Reverting to the more theoretical aspects of the paper, the diagrams shown in Figs. 2 and 6 suggest that a really stable austenite containing 18 per cent chromium requires a nickel content of at least 30 per cent. It is not entirely clear from the text what the supporting evidence is for tracing beyond 8 per cent nickel the two curves defining the ferritic and austenitic fields in Fig. 6, but some experience with the 18 per cent chrome, 14 per cent nickel alloy suggest that these curves may come down rather more steeply than shown. With carbon contents of about .06 per cent both 8 per cent and 14 per cent nickel alloys should be completely ferritic at temperatures below about 300 degrees Cent. and under conditions allowing an approach to equilibrium; the development of ferrite can, to a large extent, be brought about in the 8 per cent alloy by cold working at room temperature. Yet the 14 per cent alloy, severely cold-worked, fails to develop the slightest trace of ferrite detectible with a compass needle, and even carrying out the cold working at temperatures slightly elevated to facilitate the inversion but below the supposed inversion temperature, as 100 and 300 degrees Cent., is equally unsuccessful.

Written Discussion: By A. B. Kinzel, Union Carbide and Carbon Research Laboratories, Inc., Long Island City, N. Y.

This paper is noteworthy for its clearness of thought and elucidation of expression. The authors should be complimented on a presentation of this character. The first part, which deals with generally accepted theories,

is certainly worthy of a textbook. This part constitutes a concise review of the subject which it would be well for even those skilled in the art to read with care. In the second part, the authors tread on more debatable ground. That carbide precipitation takes place, as illustrated most aptly in their Fig. 14, is undoubtedly true in many instances, but the writer does not understand why, granted that this condition exists, it is necessary to go to theories such as those presented by Strauss and those presented by the authors to account for the intercrystalline corrosion. As the authors point out, Strauss and his colleagues believe ferrite is readily attacked, the implication being that the ferrite is high in chromium. As the authors of this paper further point out, we have innumerable instances of high chromium ferrite which is corrosion resistant. The present authors propound the hypothesis that the grain boundary material from which the carbide is precipitated is low in chromium, and that the sluggishness of the alloy is such that the chromium does not migrate. That a certain amount of migration must take place is evident from the fact that the carbides contain much chromium, and that the chromium content is probably more than can be obtained from the immediate locality. If the chromium can migrate to the extent necessary to form the carbides, it certainly should be able to migrate the short additional amount or distance needed to restore the minimum 12 per cent chromium in the grain boundary material from which the carbide has precipitated. Thus, while the hypothesis is a possible one, and in the light of present quantitative knowledge is still tenable, the writer believes that it is not probable. Anyone who has etched a quenched temperature gradient specimen of high carbon steel with no appreciable alloy content, has observed the different rates of attack. In this case, we are certain that there are only two or three constituents—ferrite, carbide and austenite, and there is little question of carbide or iron enrichment at various points. The difference in etching properties of the various microstructures has been attributed to local electrolytic effect and to local internal strain. The two are probably so intimately bound up as not to be separated at the present state of our knowledge. The writer does not see why it is necessary to go further than this very simple explanation in the interpretation of the phenomenon encountered in the chromium-nickel structures shown in Fig. 14. It is probable that the selective corrosion at the grain boundaries is due to strain and electrolytic effect increasing the rate of attack over the rate of formation of a passive film. We should certainly like to hear from the authors regarding this matter.

There is another rather interesting point in connection with this paper that might be emphasized. The authors state that the austenite can be strengthened by precipitation of the carbide, and that due to the fact that the precipitation is selective at the grain boundaries, the impairment of ductility due to this precipitation is so great as to seriously injure the quality of the material. Mr. P. P. Zeigler and the writer in experiments conducted some time ago at the Union Carbide and Carbon Research Laboratories with the alloys dealt with in the present paper, drew a tentative carbon solubility curve, as shown in the lantern slide Fig. 1. As we were

then mainly interested in the precipitation phenomenon, the curve up to 0.16 per cent carbon was not completed. This curve shows carbide resulting from quenching from the temperatures at the left of the graph. In lantern slide Fig. 2, the Brinell hardness obtained on drawing for varying periods at different temperatures is shown. It was found that the first part of the precipitation was not selective with respect to the grain boundaries, whereas the latter part was selective. Thus, by choosing a suitable time for each temperature, it was possible to obtain an alloy in which the precipitation hardening phenomenon strengthened the material without seriously injuring its ductility. For example, 62,000 pounds per square inch yield, 135,000 pounds per square inch ultimate, and 23 per cent elongation in 2 inches were obtained. It should be particularly noted that the specimens so treated were practically free from ferrite, and that the carbides were well distributed. Simple corrosion tests indicated that the corrosion resistance had been somewhat impaired, but not to such an extent as to render the material unfit for many uses. In line with the previous part of this discussion, it might be stated that this corrosion impairment took place even though there was no grain boundary effect such as shown in the authors' Fig. 14, and therefore, little chance of a chrome-poor ferrite. It should be emphasized that the immediately foregoing discussion is supplementary rather than contradictory to the work of the authors, as the carbon content of the alloys and the drawing temperature were out of the range in which they were working.

The writer wishes once more to express his appreciation of the excellent treatment of this subject given by the authors, and his hope that further experiments will clear up the moot points.

O. V. GREENE: My rather meager experience does not allow me to do any more than express a very fine appreciation of these two papers. However, I think that there is a point that ought to be presented to the authors for consideration. I believe that we are making a mistake in considering analogies between the carbides, so called, in the 18-8, and the carbides as formed in the iron carbon system, for instance. Perhaps the constituent formed in the 18-8 is not a true carbide.

Written Discussion: By O. V. Greene, Carpenter Steel Co., Reading, Pa.

The authors are to be congratulated on turning out a remarkable piece of work. The results we have obtained on these types of alloys agree with those of the authors in many respects, but differ in others. We have continued the constitutional diagram, (Fig. 1) for instance, to higher points of carbon where the solidus and A_{cm} or hypereutectoid line merge. The Ledeburite occurs at about 0.56 per cent carbon. Fig. 2 shows the structure of this constituent, austenite and the carbide enrichment in a cast sample containing 0.74 per cent carbon.

The authors' theory concerning the mechanism of corrosion in sulphuric acid and copper sulphate solutions is quite in agreement with our ideas. There is another point in the paper that some of our results may amplify. The next to the last paragraph under the heading "Carbide in

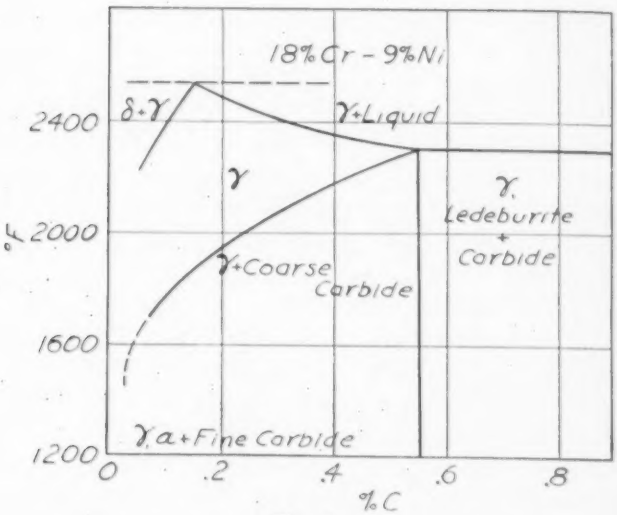


Fig. 1

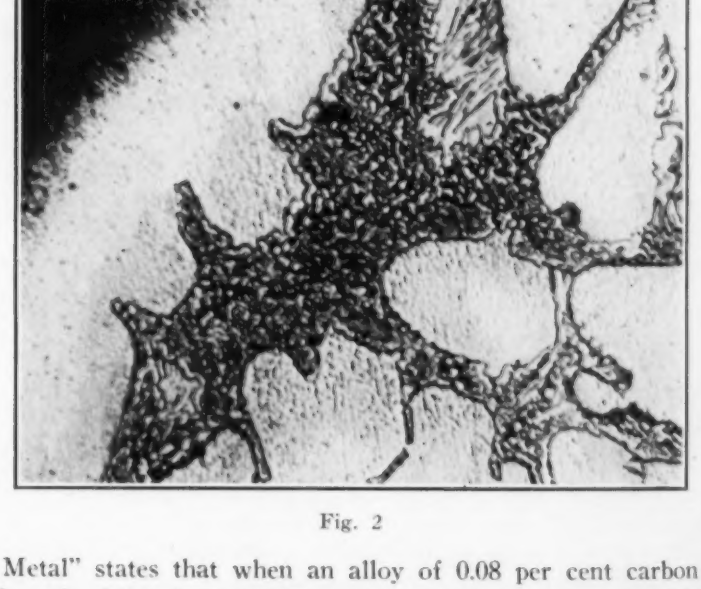


Fig. 2

Annealed Metal" states that when an alloy of 0.08 per cent carbon is heated at approximately 1600 degrees Fahr., the carbon in excess of 0.06 per cent carbon will precipitate and regions depleted of chromium will make up their deficiency from the parent grain. We find that such an alloy with this treatment will be quite immune to the attack of corrodants over long periods of time, but that subsequent heating, say at 1200 degrees Fahr., precipitates carbide again. The alloy is again in such a state that after perhaps 300 or 400 hours it will disintegrate in the acid copper sulphate. It may be that there are other variables which influence this reaction, or that it is a matter of degree, i. e., length of exposure to the acid copper sulphate. To amplify, the alloy before being heated at 1600 degrees Fahr., and merely treated by being held at 1200 degrees Fahr. might disintegrate in perhaps

less than 100 hours. However, after being given the 1600 degrees Fahr. treatment, and following it by the 1200 degrees Fahr. treatment, the immunity to attack is considerably increased.

Written Discussion: By M. A. Grossmann, vice-president and director, Republic Research Corporation, Republic Steel Co., Canton, Ohio.

The paper by E. C. Bain and R. H. Aborn, in addition to being a masterly exposition of the behaviors of stainless steels, contains at least one piece of particularly ingenious technique that calls for commendation and comment.

The study of the structures at very low temperatures has here been carried out successfully by using the method of cold working and reheating. Checking X-ray measurements against magnetic measurements furnishes convincing proof of the accuracy of their data and the plausibility of their conclusions.

The data furthermore constitute proof that what *cannot* be done by reheating alone, *can* be done by cold work followed by reheating. As the authors have pointed out, the three curves of Fig. 24 represent the behaviors of three different amounts of ferrite produced by cold work, each curve throughout its length representing the behavior of only such material in the original sample as had been transformed by cold working from austenite to ferrite.

The authors are, indeed, to be congratulated on proving their thesis so neatly and so usefully.

F. R. Palmer:[1] After two such excellent papers, I almost hesitate even to ask a question. However, there is one observation that I have made in connection with these 18-8 chromium-nickel steels that does not seem to fit into the almost perfect explanation that we have before us of their behavior.

The established facts seem to be that the 18-8 chromium-nickel type with carbon of perhaps 0.12 per cent is a super-saturated austenite and upon being heated to a favorable temperature in the neighborhood of 1000 to 1300 degrees Fahr., there is deposited in the austenitic grain boundaries a constituent generally accepted to be chromium carbide. After this deposition of carbide in the grain boundaries has been accomplished, the steel is subject to selective chemical corrosion which dissolves the grain boundaries and disintegrates the mass. It is proposed that the excess carbon migrates without its attendant chromium to the grain boundaries and there precipitates as a chromium carbide, thus exhausting the chromium content of the metal immediately adjacent to such an extent that it can no longer resist corrosion and disintegration ensues.

I have seen these alloys with precipitated grain boundaries completely disintegrated by chemical solvents so that the grains fall apart like so much loose sugar or salt. A further washing for many hours with the solvent should effectively wash from the surface of the individual grain all of the soluble material. Strangely enough, however, chemical analyses of the disintegrated grains after washing shows them to contain the same amount

[1]Assistant to the president, Carpenter Steel Co., Reading, Pa.

of carbon as the original solid metal. This experiment has been repeated several times and the results confirmed.

In light of this evidence, it seems obvious that there is little, if any, migration of carbon to the grain boundaries. This is entirely logical because we would not expect a hypereutectoid austenite soaked at a temperature below A_{cm} to deposit its carbides in the grain boundaries but would rather expect them to deposit in massive form within the grains. If such is actually the case, then any carbides precipitated in the boundaries must come from the small proportionate amount of carbon, which occurs naturally at or close to the grain boundaries. Deposition of these carbides would not disproportionately impoverish the chromium content of the adjacent metal because we know that 18-8 chromium-nickel steel (or even a simple 18 per cent chromium iron) is well able to support the precipitation of 0.12 per cent carbon and still retain sufficient chromium in the matrix to resist the attack of these disintegrating solvents. This is true even when we assume a carbide as high as Cr_4C which contains approximately 17 times as much chromium as carbon.

It would appear that we are very close to an explanation of the intergranular phenomenon observed in these 18-8 alloys but the analytical data which I have presented does not seem to fit into the theories thus far presented and I should like to know whether the authors have an explanation for the discrepancy.

O. W. Ellis:[2] These two papers scarcely need any comment. They represent a remarkable contribution to our knowledge of these systems. In regard to the first paper, I think that Dr. Krivobok's remarks as to the relatively low temperatures at which these alloys become liquid are very much to the point. I think reference to Figs. 3, 4, 5, and 6 in his paper should be made by all those who at any time have the heat treatment of these alloys under consideration, chiefly because of the profound effect of nickel content in varying the position of the peritectic point in these alloys.

I had the privilege of reading the paper presented by Mr. Bain and Dr. Aborn before the meeting. One point which impressed me very greatly was that brought out in regard to the precipitation of carbide at the grain boundaries which has been referred to by the previous speaker. Before taking that question up I should like to point out that precipitation at the grain boundaries at constant temperature is not unknown. If, for example, one takes a sample containing 58 per cent of copper, or perhaps a slightly lower per cent, heats it sufficiently, quenches it, and then reheats it at 100 degrees Cent. there is a definite increase in the hardness and a definite increase in the tensile strength. The increase in hardness and the increase in tensile strength is due to the precipitation of alpha particles in the material. If the same material be reheated to 200 degrees Cent., the hardness remains about the same, but the ductility of the material is completely changed. In fact, the material may be said to have no ductility. The reason is that alpha is now being precipitated at the grain boundaries.

[2]Director of Metallurgical Research, Ontario Research Foundation, Toronto.

Brittleness results from the very fine envelope of alpha which forms around the grain boundaries of beta. This raises a rather interesting question in regard to these iron alloys. Mr. Bain and Dr. Aborn have clearly shown (as has Dr. Krivobok) what happens when these alloys are reheated. Now, there are specifications in existence at the present time covering alloys of this type in which hardness tests are included, and in which apparently the properties of these alloys are supposed to be determined entirely by a hardness test. It seems incredible, in view of what we already know about these alloys and what now has been made so clear by these papers, that hardness tests should ever have been considered as, or possible, means for differentiating between these alloys in different conditions. Mr. Bain brings this point out very clearly.

In the typical alloy the precipitation is quite general throughout the grain, but carbide precipitation in the rustless chromium-nickel steels occurs predominately in the grain boundaries. As a result when precipitation occurs, the change in ductility is more marked than the change in hardness.

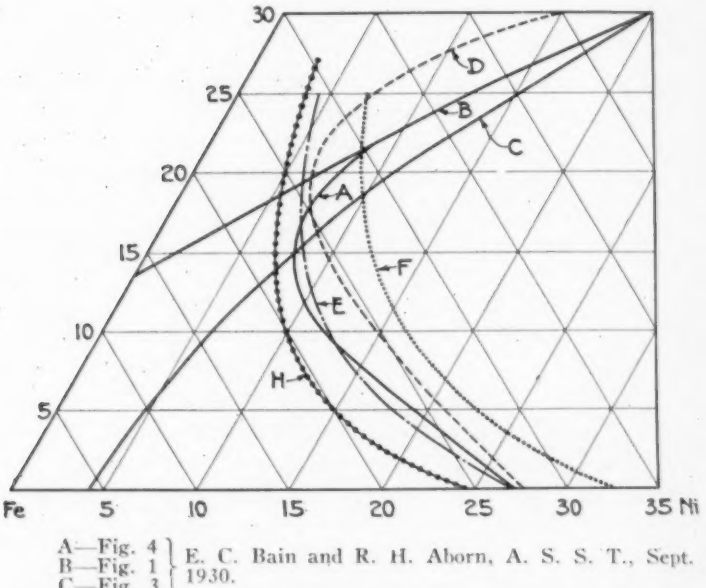
I am tempted to suggest that it would be possible to get a series of samples from one and the same alloy varying in Izod impact number from say 6 to 9 up to about 45, but all having exactly the same hardness. I myself have carried out experiments on somewhat similar chromium alloys to those described here and have found that to be true. It would be interesting if Mr. Bain could give any information along that line in respect to these alloys.

There is another point which has come to mind in looking over this paper,—referring to Fig. 7 which shows the solubility of carbon in the austenite of an 18 chromium 8 nickel alloy, there are shown 2 lines, or, rather, 2 areas, representing the precipitation of carbide. I would like to ask Mr. Bain whether he thinks it is possible, at, say, 200 or 300 degrees Fahr., under conditions of repetitive stress, for carbide precipitation to take place. Somehow or other I cannot help feeling that certain failures which I have seen might have been occasioned by precipitation of carbide at lower temperatures than those suggested by Fig. 7, due to repetitive deformation, even below the elastic limit.

P. A. E. Armstrong:[3] The papers throw a great deal of light upon what has been contended by some as being a discussion about the big and small end of the egg, which, in other words is, shall 18 per cent of chromium be employed with 8 per cent of nickel, or is it necessary to use more nickel.

A consideration of the diagrams that have appeared in prior papers and the present paper, will throw a very interesting ray of light upon what I think is an obvious situation but what has been talked about as being rather a complex one. Dr. Strauss taught that certain nickel and chromium combination was essential to make a nonmagnetic or austenitic alloy, and a good many years ago prepared a diagram, which has not been materially modified by him since, outlining just about where the alloy was alpha and where it was gamma. In substance it amounted to this that as the chromium went up the nickel went down. Strauss, I suppose, predicated this upon

[3]Consulting metallurgist, 875 Park Ave., N. Y. City.



A—Fig. 4 } E. C. Bain and R. H. Aborn, A. S. S. T., Sept.
B—Fig. 1 } 1930.
C—Fig. 3 }
D—Fig. 31. E. C. Bain and W. E. Griffiths, A. I. M. E., February 1927.
E—Fig. 1, Curve 3 } Pilling, A. S. T. M., June 1930.
F—Fig. 1, Curve 4 }
H—Fig. 1. B. Strauss, A. S. T. M., June 1924. Fig. 22—Krupp Monthly Magazine, August 1920.
All Curves Approximate.

a carbon of about 0.30 per cent, although the line that he drew was principally directed at the nickel and chromium content and not the carbon. (He said low carbon). It has been shown later that nickel and chromium with carbon, are the governing factors and that carbon is a very important element in determining the position of any dividing line.

A good many years ago, when I made chromium and nickel low carbon steels, I found that the Strauss diagram was inaccurate above about 15 per cent of chromium and concluded that the diagram was too low in chromium and nickel with low carbon because of the method of testing and the various alloys are very gradient in their change from magnetic to nonmagnetic. I found that the dividing line of nonmagnetic to magnetic approached the chromium ordinate very rapidly up to about 15 per cent and then sharply tilted in the reverse direction thereafter. Bain and Griffith in 1927 showed a line that had this general characteristic. I found that this line was inaccurate in the higher percentages of chromium and was over-generous in nickel in the lower percentages, and I am very pleased to see that the diagram of Bain and Aborn this morning puts the curve in a different position and more in keeping with the line that I had provisionally determined.

The line by Pilling, which depicts the boundary between nonmagnetic and magnetic alloys, closely follows the line of Bain and Aborn but only as far as 15 per cent of chromium, thereafter Pilling's line is as inaccurate as Strauss's original line. The Pilling curve No. 4 in his paper of 1930, based on low carbon, follows closely the general contours of the curve previously referred to. Here again curve No. 4 is inaccurate above 15 per

cent chromium if Bain and Aborn's line of today is accurate. As far as I am concerned I consider it is accurate.

Bain states that the sluggishness of the chromium-nickel alloys enables the alloy to be retained in the nonmagnetic condition when quenched down from the temperature of maximum austenite. I think Bain would have been a little more accurate if he had said the extreme sluggishness of the chromium-nickel alloys, with very low carbon, will result in the retention of the nonmagnetic phase as far as about 21 per cent chromium is concerned and 8.5 per cent nickel, but with lower percentages of chromium the sluggishness is rapidly decreased so that with low chromiums, in the order of 5 per cent or less, the alloy is not extremely sluggish, and it requires a great deal of nickel to retain the alloy in the nonmagnetic phase when quenched from the maximum austenite temperature. It is interesting to note the position of the line of Bain's curve showing the structure when the alloy has been heated to incipient fusion. It would have been instructive to have quenched the alloys from the temperature of incipient fusion and to have seen what sort of loop this would have produced.

When the 18 and 8 alloys were first produced the carbons were in the order of about 0.20 to 0.30 per cent, very few lower and quite a number higher. These alloys gave very variable results and were difficult to machine and hardened rapidly with cold work. The obvious thing was to reduce the carbon, losing sight of the fact that carbon is an important element in many of the austenitic or nonmagnetic ferrous alloys. To produce an easily worked alloy manufacturers proceeded to lower the carbon so that carbons of lower than 0.10 per cent are the order of the day. An 18 and 8, according to Strauss, is an alloy well within the austenitic or nonmagnetic region. With the low carbon, however, this is no longer true. Those manufacturers who wish to give the customer the best possible alloy are often reluctant about giving nickel but they do not mind being a little generous about chromium. On the basis, I suppose, that the more chromium the more rust resisting the alloy. They are only adding to their difficulties as Bain's line so admirably shows. It would have been a little better in some respects to have lowered the chromium about 1 to 2 per cent.

The heating of the steel for rolling has been, and probably still is, heated all over the lot. Some manufacturers feel make it good and hot, others, 1900 to 2000 degrees Fahr. is an excellent temperature. Those who are generous as to chromium and heat find themselves in the lefthand side of Bain's incipient fusion curve, and the 18 and 8 with low carbon becomes an alloy that in some parts is nonmagnetic and in others slightly magnetic. If the steel is to be made hot during working then 18 chromium should certainly have at least about 12 per cent of nickel. And if 8 per cent of nickel is to be employed and the alloy made as carefully as it can be made, and heat treated to the maximum austenite temperature, then you can produce an alloy that is austenitic and nonmagnetic just as long as you do not hit it. If the heat treatment is carried too high the alloy will not be entirely nonmagnetic and, particularly is that so, when the heat happens to run nice and low in carbon and the chromium a little on the high side.

This has been extremely well set forth in Krivobok and Grossmann's paper.

To try and improve 18 and 8, by adding to it elements, that tend to retain iron in the alpha phase is not going to help the situation, in fact, makes it worse. For instance adding manganese to such an alloy, if the carbon is very low, will not make the alloy more stable austenitic but will actually have the effect of making the alloy more magnetic. The same is true with tungsten or molybdenum. It so happens that if manganese is alloyed with chromium iron, carbon less than 0.10 per cent, it is practically impossible to produce a nonmagnetic alloy with any chromium content, certainly over 10 per cent. Although I was able to produce a nonmagnetic alloy, but only one, which contained 20 per cent of chromium and 60 per cent of manganese, carbon low. This alloy was as brittle as the proverbial pipe stem. On the skin file hard and the underlying portions filable, but nonmagnetic both as to the skin and inside portion when tested by a magnetised needle a half inch long and very freely suspended. This needle by the way is sufficiently sensitive to pick out the north and south poles on a hammer bruised sample of 18 and 8. Not a highly scientific piece of apparatus but very informative and extremely sensitive.

It is probable that we shall have the 18 and 8 type alloy with still lower carbon, and in the days of the high frequency furnace carbon is not necessarily an accompaniment of melting. Incidentally the machinability of an 18 per cent chromium and 12 per cent nickel alloy is much better than one containing 18 per cent chromium and 8 per cent nickel, the carbon in both instances being about 0.07 per cent. Very low Brinell can be obtained with higher nickel. The malleability and cold deforming qualities of 18 per cent chromium, 12 per cent nickel, with very low carbon, is, in my opinion, superior to that of the same low carbon analysis but with 8 per cent nickel.

In view of the nature of the disclosure in both of these papers and the teachings, it is clear that 18 and 8 should not be adhered to merely because that was the early type analysis but it should be amended to take care of the low carbon effect. There may be a few instances where 18 and 8 is better suited than 18 per cent chromium and 12 per cent nickel, with very low carbon, but for the majority of applications the change can be made without anybody being inconvenienced. If higher nickel is desired 18 per cent chromium and 16 per cent nickel, and 18 per cent chromium and 20 per cent nickel, are all very good alloys and anywhere between 12 and 16 per cent nickel will meet the very great majority of applications.

It is interesting to look at Bain's quenched diagram and see that 5 per cent chromium requires about 20 per cent of nickel to make the alloy nonmagnetic and austenitic, and it requires the same amount of nickel to make a 30 per cent chromium alloy austenitic. This is, of course, very obvious because of the tremendous effect chromium has upon iron in maintaining the alpha phase. After all, what else can you expect, chromium is body-centered cubic and so is alpha iron. It is true, iron can exist as face-centered cubic but not so chromium. Therefore, the higher the chromium,

in the absence of carbon or nearly so, the more stable is the alpha phase, and the enormous effect of carbon in retaining the gamma phase is readily appreciated when it is borne in mind that about 22 per cent of chromium requires about 0.50 per cent carbon to make it nonmagnetic when quenched from the temperature of maximum austenite, whereas if very low carbon is employed and nickel is used as a substitute to produce gamma iron then between 9 and 10 per cent nickel is required, and if it is made over hot (incipient fusion) then some 13 per cent nickel is required.

It seems to me that safety factors should not be ignored. In the days of low priced automobiles and airplanes we run safety factors of one and sometimes less, but in alloys of the chromium-nickel type a safety factor is an important thing. When it is less than one the alloy is not what you hoped it to be. If the factor safety is only one it is a dangerous thing, as a general expression, to adopt this as a standard when a phase change can be brought about, not by heat essentially, but by slight mechanical disturbance. If two parts are put together it generally means mechanical work, particularly if the structures are of any magnitude. Therefore the factor of safety must be some multiple of one.

It has been suggested that 18 and 8 has become standardized and therefore why institute a change. I feel that it is not 18 and 8 that has become standardized but that this type of material has become a standard one in which to design, and simply because we had carbon tool steels for years and they were very satisfactory and still are, that is no reason for not using high speed steel or alloy tool steel.

So as to help my discussion, and perhaps make the various points I have raised clear, I have prepared a diagram setting forth the various curves referred to superimposed one upon another. This I have found very helpful and I trust it may be of some help to others.

T. H. NELSON:[4] I should like to take this opportunity of endorsing much that has been said, particularly by the author of the last paper. I feel that at no time during the development of stainless steel have more comprehensive papers appeared on this subject. The last few years my work has been substantially in the practical field rather than the laboratory and I find it is often a fact that "Works practice" brings to light problems and often the solution of this same problem before the metallurgist is able to present the solution, and this I believe to be emphasized substantially in these papers this morning.

Mr. Griffith stressed substantially the question of standardization and when he made mention of the fact that some of the information presented by Mr. Bain could hardly be compared to general practice I think Mr. Griffith must have overlooked the direct relation between the graduated heated specimens shown by Mr. Bain and its particular applicability to the problems giving serious cause for investigation in the field of welding these alloys. In welding we have exactly the condition illustrated by Mr. Bain on his graduated test piece, for we have all conditions from that of the liquid metal being laid in the weld itself to material at normal temperatures

[4]Consulting Metallurgist, Midvale Co., Philadelphia.

as the heat is dispensated away from the welded area and within this range the possibility of many problems arising.

The reason I am at the moment somewhat doubtful of the possibility of standardization on 18 and 8 is this very tendency and the fact that I believe alloys better adapted to the art of welding are within sight.

We all agree that the steel maker and user for that matter of fact would be glad to find one article that would fill all the requirements of a corrosion resistant metal. Personally I believe that far from being in a position at the moment to standardize we are confronted with the production of a series of alloys which will be either chromium-nickel alloys with iron, or nickel-chromium alloys with iron according to the corrosive condition to be met. There are many instances where nickel is the element substantially resistant to corrosion and to these alloys it is possible to add considerable percentages of chromium to adjust the physical properties without seriously impairing its corrosion resistance. On the other hand there are conditions where chromium is the element that offers resistance to corrosion and to these alloys it is possible to add nickel to adjust the physical properties without seriously impairing the resistance of the ultimate alloy to corrosion; not in anyway losing sight of other elements such as silicon, tungsten, molybdenum, etc. With this thought in mind I feel we are far from the possibility of standardizing and rather that our work so far has simply opened the way to the development of many alloys for the varied corrosive conditions to be found throughout the various industries.

I congratulate the authors on their various papers. I believe the papers have a very wide and broad interpretation but I should like to see, particularly in the A. S. S. T., where we have always prided ourselves upon the fact that we embraced the practical man and "the man at the fire" so to speak, that the theories and evidence contained in our technical papers should be carried forward wherever possible to their practical application, otherwise much valuable data becomes lost in the archives of technical literature.

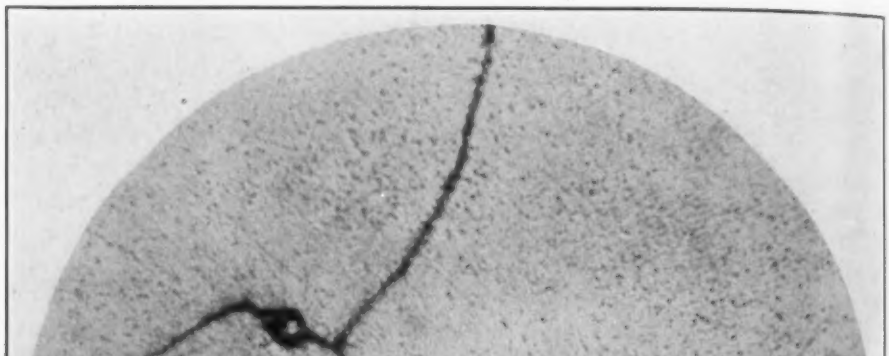
Authors' Closure (Dr. Krivobok)

Only a few men have as intimate knowledge of chromium-nickel alloys as Mr. Palmer. It was, therefore, a real pleasure to hear from him. The question raised in Mr. Palmer's discussion is, indeed, a most pertinent one: truly, the question of precipitation is no longer of academic or passing interest. Unless we understand the principles underlying its process and divulge its nature, we shall always be in fear of similar phenomenon in these alloys as well as in the others,—not necessarily, of course, under identical conditions. The very nature of these alloys calls for service under, shall I say, unusual conditions and is it not true that in most cases we are, as it were, feeling our way in darkness? Far be it from me to attempt to give the correct answer to Mr. Palmer's query. A few thoughts on the subject may perhaps be allowed.

I am inclined to believe, regardless of some seemingly contradictory findings, that visible disintegration at the grain boundaries, also in other definite places, such as twinnings, of "18-8" is of the nature of carbide pre-

cipitation. Naturally one would expect, as Mr. Palmer suggests, that a careful chemical analysis of the collected precipitate will settle the question. It is not to be forgotten, however, that such an analysis is quite difficult: precautions must be taken that the analyzed precipitate does not contain small pieces of "18-8" proper. My esteemed co-author, Dr. Grossmann, wishes me to add (and I, indeed, fully agree with him) that the migration of carbon to the grain boundaries, to which Bain and Aborn referred, and to which we also referred, is a migration of carbon in the intergranular areas only. That is, only very narrow bands immediately adjacent to the grain boundaries are depleted of carbon. It is these narrow bands which are eaten out by the etching reagent. The rest of the grains keep their carbon, because the diffusion is so sluggish that the carbides would not reach the grain boundaries in any reasonable time. It is for this reason that the grains themselves, being unaffected, show their original carbon content. The only depletion is in the narrow bands at the grain boundaries. Preliminary work,—I refer to it as "preliminary" because I do not think it is altogether conclusive,—carried on on "18-8" with excessively large grain size, after very long disintegration period, *did show* a much higher carbon percentage, as compared with carbon in the steel proper, but not sufficiently high, however, to correspond to any one of the chrome carbides, even of the lowest carbon, such as Cr_4C, mentioned by Mr. Palmer.

I hold,—and my observations can easily be substantiated, that carbide precipitation is *not* confined *only* to grain boundaries, but takes place throughout grains as well (see accompanying photograph); quite true, it is not anywhere near as pronounced as at the boundaries (or at the slip planes) but it can, nevertheless, be plainly observed. The intensity of precipitation at grain boundaries and slip planes brings forcibly to mind the importance of internal stresses. I wonder if it would be in order to bring this matter to the attention of the reader? We hold that "18-8" or, for that matter, any other chromium-nickel steel, if you wish, can be rendered austenitic in nature, by adding sufficiently high percentages of the mentioned elements. That is to say, with "18-8", it is chrome-nickel-carbon solid solution (chromium-nickel austenite) preserved at room temperature. Although it is related in many respects to the austenite of carbon steels, we can not, or, I would like to say, we must not forget, that carbon steel austenite is a high temperature phase, stable within the definite high temperature region, rarely and with much difficulty preserved at room temperature, and most certainly not intended for service at ordinary temperatures at which it is easily decomposed. The phase known as chromium-nickel austenite (which we find in our chromium-nickel alloys) is stable at high temperatures, also easily preserved at room temperature. We cannot hold, however, that the retention of austenite at room temperature is a criterion for its being a stable phase at that temperature. In the case of *stable* austenite, we could not possibly affect its decomposition by raising the temperature: to claim so would be equivalent to discarding the principles of phase rule. Since we do decompose this phase,—not only in chromium alloys but in others as well,—we have to accept the other alternative, namely, that in most austenitic



alloys we are dealing with conditions of metastability.* Once this postulate is admitted, the occurrence of the phenomenon of decomposition and carbide precipitation becomes much less involved, for any factor, such as internal stresses, for example, would have a tendency to destroy metastability. Furthermore, heating,—provided it is confined to temperatures within the range of metastability,—would further tend to decompose the metastable phase.

Our knowledge of internal stresses and their concentration is, indeed, a meager one. Is it out of order, however, to speculate that we have higher stresses at grain boundaries due to the discontinuity in crystallographic orientation? In such a case,—based on speculation, I admit, but justifiable,—decomposition is likely to occur at grain boundaries, or, as I said before, at any other place where the stresses are made, by nature or by artificial means, sufficient to provoke decomposition.

I am pleased to hear again from my esteemed friend, Dr. Ellis: his remarks are always most pertinent and stimulating. I am glad, naturally, that he shares my views in regard to the position of the peritectic reaction (termed.

*Incidentally, it might be pointed out that the studies of metastable ranges on the constitutional diagrams of the alloys would, probably, yield much valuable information.

in the paper, as the appearance of "liquefied" phase). I have not at present any careful data on Izod impact figures, but have done some preliminary bending tests: it will be observed from the paper that the hardness of the alloys is rendered *gradually* lower as the quenching temperature is increased: from 2200 degrees Fahr. to 2400 degrees Fahr., hardness remains practically the same but bending tests show a marked change in bending ability in the 2400 degrees Fahr. treated alloy, which has hardly any ductility at all. I believe that a similar observation will be obtained on testing nickel-chromium steels which have undergone low temperature decomposition. I am equally certain that repetitive deformation, as Dr. Ellis terms it, would result in carbide precipitation at all kinds of temperature.

Dr. Grossmann and myself fully agree with Mr. Armstrong's remarks that heat treatment carried to too high a temperature will develop delta-iron if carbon and nickel are too low or if chromium is too high. Dr. Grossmann kindly reminds me that this is shown, in addition to the microstructure, in the magnetic values of Fig. 31 where quenching temperatures above 2300 degrees Fahr. develop the magnetic constituent. Gladly we agree with Mr. Armstrong that Bain's and Aborn's work also proves this quite conclusively.

Mr. Armstrong's clear discussion is, indeed, a worthy contribution to the admirable piece of work on the part of Aborn and Bain. I hope that others interested in chromium-nickel alloys will not overlook the fact that the heat treatment of such alloys must not be run at too high a temperature.

Mr. J. J. Kanter has kindly raised the question regarding the relations between the carbon content and the magnetic flux density of the alloys after such alloys have been disintegrated by heating to 1200 degrees Fahr. The curve for the "18-8" alloy containing 0.14 per cent carbon shows, as stated in the paper, lower flux density than an alloy with 0.046 per cent carbon. A very pertinent question is asked by Mr. Kanter: If magnetic flux density is entirely due to carbide precipitation, would it not mean then that an alloy with lower carbon content is less stable than one with higher carbon content? I would like to point out in this connection that in case of 0.046 per cent carbon, we have an alloy which, on heating to high temperatures, would be located on the diagram (see Fig. 5, tenth page) very close to the dividing line between "austenite" region and "delta-austenite" field. In other words, the magnetic characteristics of the alloys very low in carbon will depend upon the true position of the above-mentioned line. It might also be, as Dr. Grossmann reminds me, that alloy 804 *as a whole* is so unstable that reheating causes considerable break-down of the austenite with a consequent appearance of the magnetic alpha iron.

I wish to thank, most sincerely, Dr. Aborn for his kind remarks and his constructive discussion. There is no doubt whatsoever that coarse agglomerated carbides go into solution with great difficulty. Our treatment (several hours at 1800 degrees Fahr.) was really a final of the several preliminary treatments, if I may use such a clumsy phrase, immediately preceding quenching of the alloys for the study of the diagram. Previous to 1800 degrees Fahr. treatment, our alloys were heated and forged several

times. The possibility that some of the larger carbides still remained, as such, is, of course, admitted. For this reason, I am quite willing to accept that the results of Dr. Aborn and Mr. Bain regarding the probable course of the carbide solubility curve are more accurate. The hardness of the same alloys, however, will still show the same downward trend, as I was able to find out, after reading Dr. Aborn's kind letter, even if they were subjected to prolonged treatment, such as, for example, twenty hours at temperatures between 1800 degrees Fahr. and 2300 degrees Fahr. Above that temperature, the hardness results are quite erratic due to the appearance of the liquefied phase.

It was, indeed, most interesting to observe the very definite upward trend of the magnetic curve as is shown in Fig. 31 of the paper. It was particularly pleasing because it enabled us to place approximately, the first appearance of the magnetic delta phase. I am, however, very much concerned about our failure to observe similar upward trends in the "18-8" alloys of high carbon composition. I have always held that liquefied phase is the result of the peritectic reaction and that, as a consequence of this reaction, some delta phase should be formed; in fact, I am reasonably certain that I observed it under the microscope. The magnetic curves, however, do not indicate higher magnetic induction curves after quenching from this high temperature. I am, indeed, appreciative of Dr. Aborn's suggestion in regard to the shifting of the magnetic induction curves toward lower values if the heating time at quenching temperature is increased. I have not been able, as yet, to carry out this suggested investigation but, indeed, intend to do so.

In reading over the written discussion by Dr. Kinzel (of Dr. Aborn's and Mr. Bain's paper) I was particularly pleased to note that he does not overlook the possibility of local internal stress.

In closing my lengthy reply to those who so kindly participated in the discussion, I would like to make one more remark. I do not believe that the time has come when we could safely say that "18-8" is *THE* alloy for *ANY* purpose; neither am I willing to advocate that we ought to discard "18-8" and seek another alloy merely because "18-8" did not fulfill the expectations of a careless individual. To my mind, the most dangerous tendency in regard to special alloys is a lack of intelligent and critical consideration of the conditions under which such alloys are supposed to serve. Is it not obvious that without such consideration, we are no better off than in those days when special alloys were unknown?

I wish to thank all of the gentlemen who have pleased us by participating in the discussion.

Authors' Closure (E. C. Bain)

The discussions offered in connection with our paper are from such maturity of view-point that we derive much encouragement from them. Especially is this true when we consider that our effort has elicited the expenditure of the time and care in preparing discussions of such excellence and understanding. We, therefore, wish to thank all who have contributed to this entertaining discussion. If the paper is not worthy of preservation on its own account it should, nevertheless, be regarded as worth while when

it carries with it these thoughtfully critical comments. If we may be permitted to do so, we should like to mention our appreciation of the paper by Krivobok and Grossmann on a similar subject presented on the same occasion. It has been extremely interesting to us.

Mr. Palmer's report of the ingenious experiments on the analysis of the disrupted grains collected after complete inter-granular attack constitute a valuable contribution to this subject. From the microscopic appearance alone of the "sensitized" metal one would be certain that the total carbon removed from solution to form carbide would be an inconsequential part of the whole carbon content. However, thin as the non-resistant film between the grains may be, the loss in chromium by carbide precipitation seems to demand that carbon was drawn from a somewhat greater distance than the chromium, and if so, it is, after all, only to be expected that carbon diffuses more rapidly than chromium. The lowering of chromium content required in the loss of corrosion resistance is so great as to imply that carbon has been transferred from a greater distance than the chromium. Our own measurements of loss in weight of specimens undergoing the intergranular attack indicates that the "sensitive" metal is too small in mass to estimate, and the actual loss in weight is not consistently greater in metal showing carbide precipitation than in normal annealed metal. We believe that precipitation of excess constituents, when the supersaturation is not very great, occurs by preference in the disorganized (or amorphous) metal of the grain boundary. In this case the precipitation is amazingly minute in the early stages but yet sufficient to render the metal very easily attacked.

Dr. Ellis' remarks form a valuable extension of this paper. In our opinion, the transformation of some of the austenite to ferrite in a form resembling martensite is the principal accompaniment of stress at low temperature upon the austenitic alloys here discussed. However, it is clear that carbide precipitation may proceed at a lower temperature in cold-worked metal than in well crystallized metal. We observed that some slight precipitation occurs after hundreds or thousands of hours at temperatures as low as 900 degrees Fahr. in the cold-worked metal, in a form, however, which does not entail the serious inter-granular depletion. We have no information indicating what the effects of repeated stressing at moderate temperatures may be.

With most of the entertaining discussion by Mr. Armstrong we find ourselves in agreement, and we appreciate particularly the emphasis he has placed on some of the points we have had to refer to only briefly. While it is now clear, through the recent German publications, that manganese is by no means the great stabilizer of austenite that general opinion formerly indicated, it is, nevertheless, in the same general class with nickel and therefore not in the same class as tungsten, molybdenum and chromium. Perhaps it is because of the Hadfield manganese austenitic steel that the impression was created that manganese is the outstanding element for lowering the transformation of iron, but in this steel the carbon is usually very high. Actually, in the presence of chromium and low carbon it is distinctly less the austenite stabilizer than nickel, and furthermore appears

to contribute less sluggishness. The remarks relating to the adjustment of the nickel content to suit the application are very timely.

We wish to thank Mr. Nelson for emphasizing the identity of our temperature gradient specimen with the conditions arising in welds. The necessity for brevity has prevented many references to the close analogy between our experimental conditions and those found in the utilization of the rust-resisting alloys, but in general we have tried to discover the fundamental metallurgical nature of these alloys by methods which had some counterpart in engineering applications.

Mr. Pilling's remarks serve the very important purpose of stressing the profound effect of nickel to reduce the amount of ferrite (and attendant abnormal hardening) accompanying cold deformation. Indeed, in the ordinary sense, nickel present to the extent of perhaps 15 per cent as pointed out by Mr. Pilling reduces such transformation almost to a theoretical consideration. However, this effect appears to us to be a case of rendering austenite unusually persistent but not stable. As an example, extensive investigation upon a 20 per cent chromium, 15 per cent nickel alloy shows the development of a very appreciable amount of ferrite even though the carbon content was as high as 0.18 per cent. The lower carbon alloys are correspondingly more easily rendered ferritic. Again, an alloy of 0.03 per cent carbon and 13 per cent nickel develops cold work ferrite when only 14.5 per cent chromium is present. The lower chromium alloys are distinctly less liable to contain the stable ferrite. The lines as drawn in our diagram might perhaps best be shown dotted at the higher nickel contents, but we are of the opinion that with negligible carbon the result would be substantially as indicated under the condition of equilibrium, a condition not likely to be realized in any ordinary method of treatment. As to behavior in use and in fabrication we feel certain that we are in substantial agreement with Mr. Pilling.

Mr. Kinzel makes several very rational suggestions as to the nature of the various carbide precipitation phenomena. Our theory includes a more complex concept of the phenomenon only because of certain observations which cast doubt upon the more simple explanation by electrolytic action. The factors which we believe pertinent are: (1) the extreme thinness of the attackable paths between the grains; (2) the full resistance of the grains themselves to attack; (3) the infinitesimal mass of the carbide actually precipitated; and (4) the improbability of the electrolytic action occurring. Only the last need be extended. Samples of the chromium rustless alloys, into which fragments of gold were rolled, showed the same resistance to mild attack in the vicinity of the gold cathode particle as elsewhere. The regular electrolytic action then seems to be obscured somewhat by the passive surface condition which is generally assumed to be responsible for the high resistance of the chromium alloys in an oxidizing environment. It appears to us that a precipitation of carbide particles *per se* would not so greatly increase the local corrosion rate unless the protective film were also inhibited (or incomplete) and this, in turn, demands a composition change.

It will be extremely interesting to form some classification of the

modes of separation of excess constituents from supersaturated solid solutions. The work of Dr. Mehl at the Naval Research Laboratory appears to be taking such a course although it was begun rather as a study of only one of these modes of separation, the Widmanstätten structure. It is a thoroughly worth while study and Dr. Mehl's first papers show that the question will be adequately treated.

Mr. Greene makes some observations which would enable us to complete the carbon diagrams to the eutectic (or ledeburite) line. As would be expected his information indicates that the increase of carbon solubility near the melting point is very rapid. It appears that carbide precipitation within the temperature range of chromium migration and hence also abundant coalescence of carbide does not result in intergranular sensitivity, but the temperature for rapid coalescence is high enough definitely to dissolve more carbon. Carbide thus dissolved in excess of the solubility at 1200 degrees (for example) may be reprecipitated at the lower temperature with ultimate intergranular sensitivity. However, the time factor is important and permits the partial desensitization at least.

STUDY OF HIGH-CHROMIUM, LOW-CARBON STEEL*

ARTHUR PHILLIPS AND RALPH W. BAKER

Abstract

The first section of this paper includes a series of experiments showing in a semi-quantitative manner the rate of grain growth in a heat-resisting steel of 28 per cent chromium and 0.25 per cent carbon. Charts accompany to graphically correlate factors of time and temperature with grain size. The experimental evidence leads to the conclusion that in this steel the grains reach their maximum size after comparatively short periods at high temperatures.

The second part of the paper is confined to the study of the micro structure of steel containing approximately 28 per cent chromium and carbon contents ranging from 0.07 to 0.26 per cent. With reference to the structural changes and the effect of carbon on the alpha-gamma and gamma-delta points, previous investigations have placed the maximum carbon content for gamma-free alloys at about 0.20 per cent. The experimental results, discussed in this paper, place the limit at approximately 0.10 per cent carbon.

ONE of the important advances in ferrous metallurgy of the last decade has been the development of a number of alloys specifically conspicuous for their corrosion and heat resisting properties. The present paper deals with a study of a high chromium-low carbon steel which is popularly reported to be permanent in oxidizing atmospheres up to about 2100 degrees Fahr. (1150 degrees Cent.) and, for many purposes, may be safely used up to 2235 degrees Fahr. (1225 degrees Cent.). Unfortunately this alloy containing approximately 28 per cent chromium and 0.25 per cent carbon, at the higher temperatures, is subject to rapid grain growth; and the extremely coarse structure developed is responsible for a

*From an essay presented by Ralph W. Baker to the Faculty of the Graduate School of Yale University in partial fulfillment of the requirement for the Degree of Master of Science.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. Of the authors, who are members of the society, Arthur Phillips is Associate Professor of Metallurgy, Yale University, New Haven, Conn., and Ralph W. Baker is Research Metallurgist with the Republic Steel Corp., Canton, O. Manuscript received May 14, 1930.

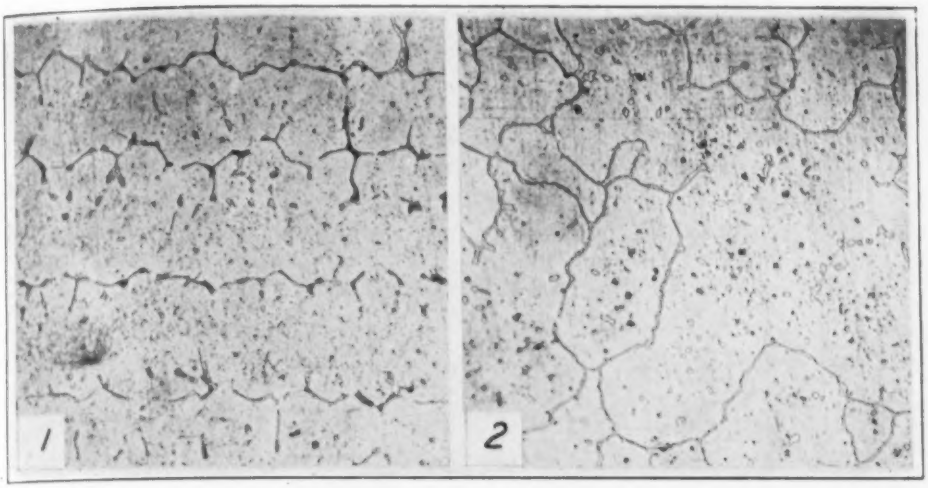


Fig. 1—Cast Structure. Chromium 27.82 Per Cent, Carbon 0.26 Per Cent. Fig. 2—Cast Structure Homogenized. Both Specimens Etched with Aqua Regia in Glycerine. × 100.

degree of brittleness which precludes its use for many purposes for which it is otherwise well adapted.

The first section of this paper includes a series of experiments showing in a semi-quantitative manner the rate of grain growth in this steel at several temperatures for periods ranging from 4 to 20 hours. The second part is confined to the study of the microstructure of steels containing approximately 28 per cent chromium and carbon contents ranging from 0.07 to 0.26 per cent.

Grain Growth Experiments

For the grain growth work a five pound ingot of the following composition was prepared: chromium 27.82 per cent, carbon 0.26 per cent, silicon 0.34 per cent, manganese 0.39 per cent. It was melted in a 35 kilovolt-ampere high-frequency induction furnace, using Armco iron and low carbon ferrochromium as the base materials. After homogenizing the casting by annealing for several hours at 1650 degrees Fahr. (900 degrees Cent.) followed by furnace cooling, seven pieces were cut from the central part of the ingot. Each specimen was then given a 20 per cent reduction in thickness by cold working. Figs. 1 and 2 are representative of the cast and annealed structures. A discussion of the structural elements present in this alloy is included in the second section of this paper.

One specimen was used for each of the temperatures chosen for the annealing schedule. After annealing at a given temperature for four hours the sample was furnace cooled, ground and polished, and a grain count made. It was then annealed for another four hours at the same temperature and a second grain count obtained. This procedure was followed until each specimen had been annealed

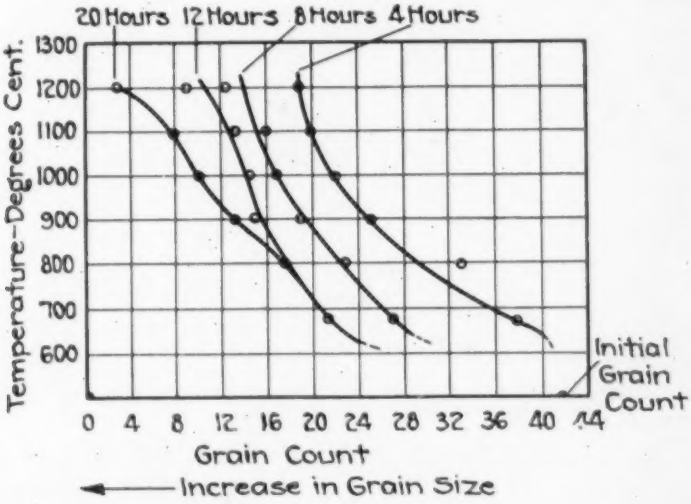


Fig. 3—Grain Count Curve.

for a total time of twenty hours. In other words, this method permitted the examination and grain count of each specimen for cumulative annealing periods up to and including twenty hours. The temperature range and specimen designations are given below:

Specimen Designation B. G.	Corresponding Temperature of Anneal	
	Degrees Fahr.	Degrees Cent.
1	1245	675
2	1470	800
3	1650	900
4	1830	1000
5	2010	1100
6	2190	1200
7	2370	1300

The grain counts were obtained by counting every grain included within a rectangular area, 3¼ by 4¼ inches, outlined on a ground glass plate. With the exceptions of the specimens annealed at the highest temperatures a magnification of 100 diameters was used; in the case of the extremely coarse grained structures a magnification of 10 diameters was employed and the counts so obtained were evaluated in terms of the higher magnification.

Fig. 3 correlates the factors of time and temperature with grain size in isothermal curves in which grain count as abscissa has been plotted against time in hours (cumulative anneal) as ordinate. Time and temperature with grain growth for equal periods of anneal are plotted in Fig. 4. Photomicrographs, Figs. 5, 6, and 7, show the structures resulting from the 8, 12, and 20 hour anneals at 2190

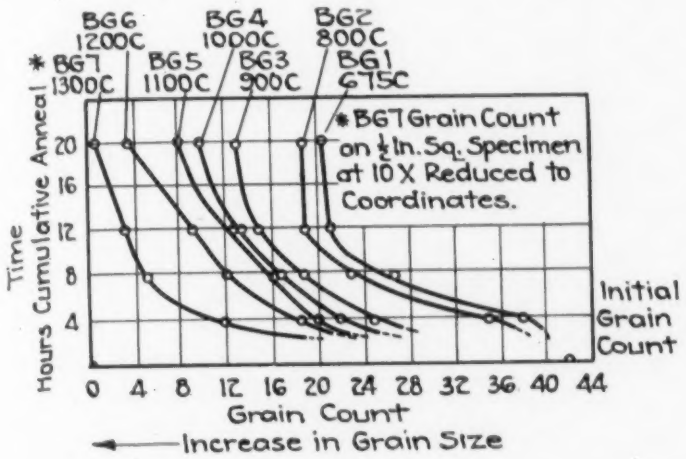


Fig. 4—Grain Count Curve.

degrees Fahr. (1200 degrees Cent.) and they may be regarded as typical of the structural characteristics of the whole series of heat treatments. Attention is called to the fact that the structures resulting from the prolonged annealing are free from austenite only by virtue of the considerable decarburization produced by the several hours at elevated temperatures.

The experimental evidence leads to the conclusion that steel of this composition is subject to relatively rapid grain growth at the lower temperatures 1290 to 1830 degrees Fahr. (700 to 1000 degrees Cent.) and to extremely rapid growth at higher temperatures 1830 to 2550 degrees Fahr. (1000 to 1400 degrees Cent.) Furthermore, the grains approach their maximum size after comparatively short periods at elevated temperatures. Fig. 8 shows the microstructure of a pyrometer tube (chromium 28 per cent carbon 0.26 per cent) which had been heated intermittently for three months at 1560 degrees Fahr. (850 degrees Cent.). Its grain size may be compared with that of Fig. 7 developed by 20 hours at 2190 degrees Fahr. (1200 degrees Cent.) and Fig. 9 produced by heating for

seven days (168 hours) at 1650 degrees Fahr. (900 degrees Cent.). This observation regarding the rapid grain growth of this alloy is in agreement with the experience of mill operators who report that it develops extremely coarse grains during short annealing periods at moderately high temperatures. In processing this steel it is important, therefore, to use the lowest effective temperature in order to avoid the brittleness associated with the coarse grained structure.

Microstructures of Alloys Containing 28 Per Cent Chromium and 0.07 to 0.26 Per Cent Carbon

As far as is known Bain[1] was the first to develop and preserve at ordinary temperatures the delta iron solid solution, although Westgren and Phragman[2] had previously determined by X-ray studies that the alpha and delta forms are identical (body-centered cubic). Bain's experiments, which have been confirmed by Esser and Oberhoffer[3] and others, showed that the gamma transformation of very low-carbon iron may be suppressed by the addition of 13 to 14 per cent chromium. In other words, in this alloy the body-centered form of iron remains unchanged from low temperatures up to the melting point.

It was also clearly indicated by Bain's work that the minimum chromium content necessary to suppress entirely the gamma change is influenced by the carbon content. Carbon tends to extend the region of gamma stability into the higher chromium field. Furthermore, carbon lowers the alpha-gamma transformation temperature and raises the gamma-delta point.

This section of the paper deals with a study of steels containing 28 per cent chromium and 0.07 to 0.27 per cent carbon. In discussing alloys in this class Bain states that "this group (25 to 35 per cent chromium) is characterized by being dominantly without transformation. The major portion of the alloy, even with carbon up to 0.35 per cent, remains unchanged throughout the entire temperature range between room temperature and the melting

[1]E. C. Bain, "The Nature of Alloys of Iron and Chromium", Transactions, American Society for Steel Treating, Vol. 9, 1926.

[2]Westgren and Phragman, *Journal*, Iron and Steel Institute, 59 (1924) 159.

[3]Esser and Oberhoffer, *Stahl und Eisen*, 47 (1927) 2026.

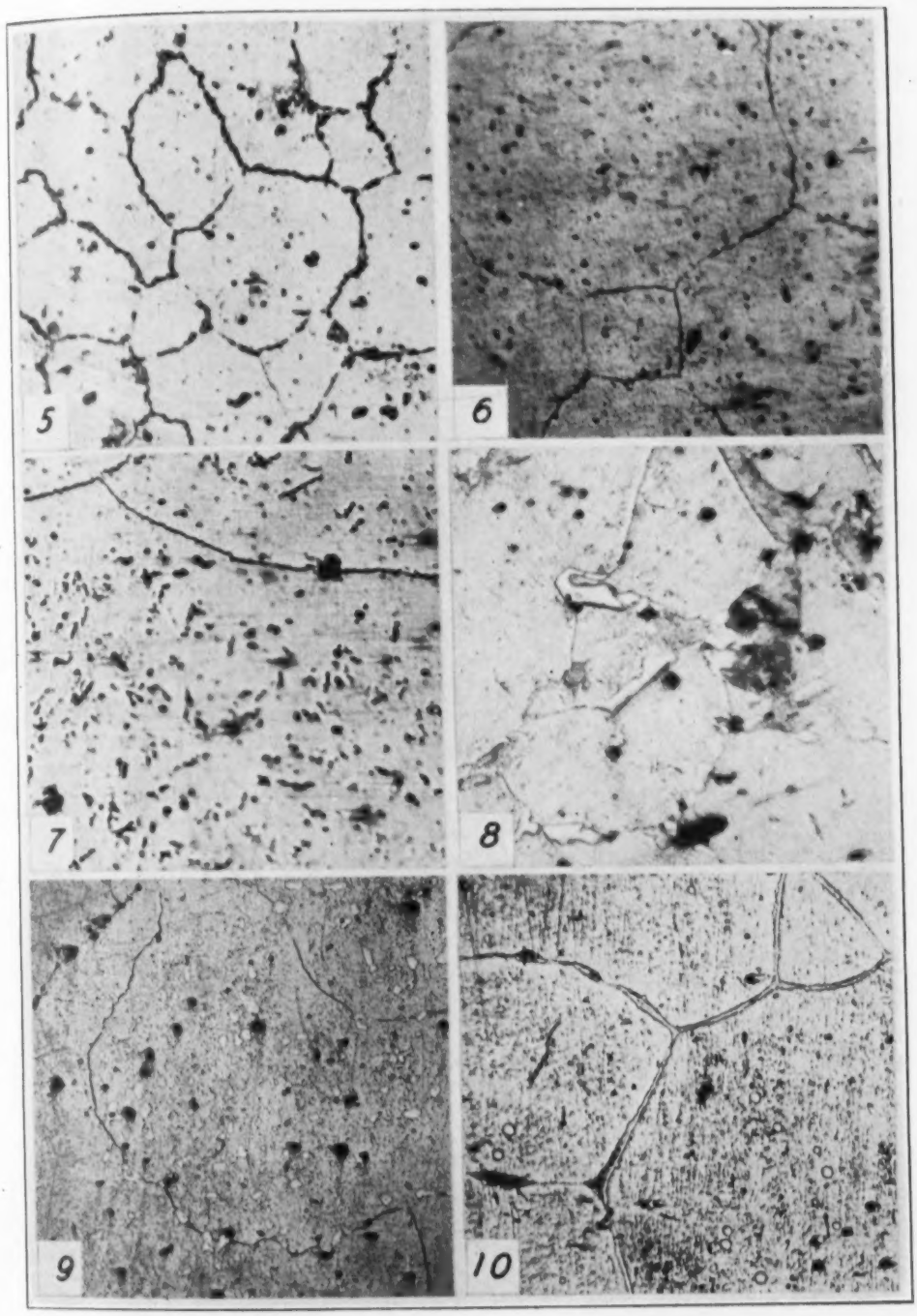


Fig. 5—Annealed 8 Hours at 1200 Degrees Cent. (2190 Degrees Fahr.). Fig. 6—Annealed 12 Hours at 1200 Degrees Cent. (2190 Degrees Fahr.). Fig. 7—Annealed 20 Hours at 1200 Degrees Cent. (2190 Degrees Fahr.). Fig. 8—Pyrometer Tube End. Fig. 9—Annealed for 168 Hours at 900 Degrees Cent. (1650 Degrees Fahr.). Fig. 10—Chromium 28.81 Per Cent, Carbon 0.16 Per Cent. Quenched after 2 Hours at 1300 Degrees Cent. (2370 Degrees Fahr.). All Specimens Etched with Aqua Regia in Glycerine. × 100.

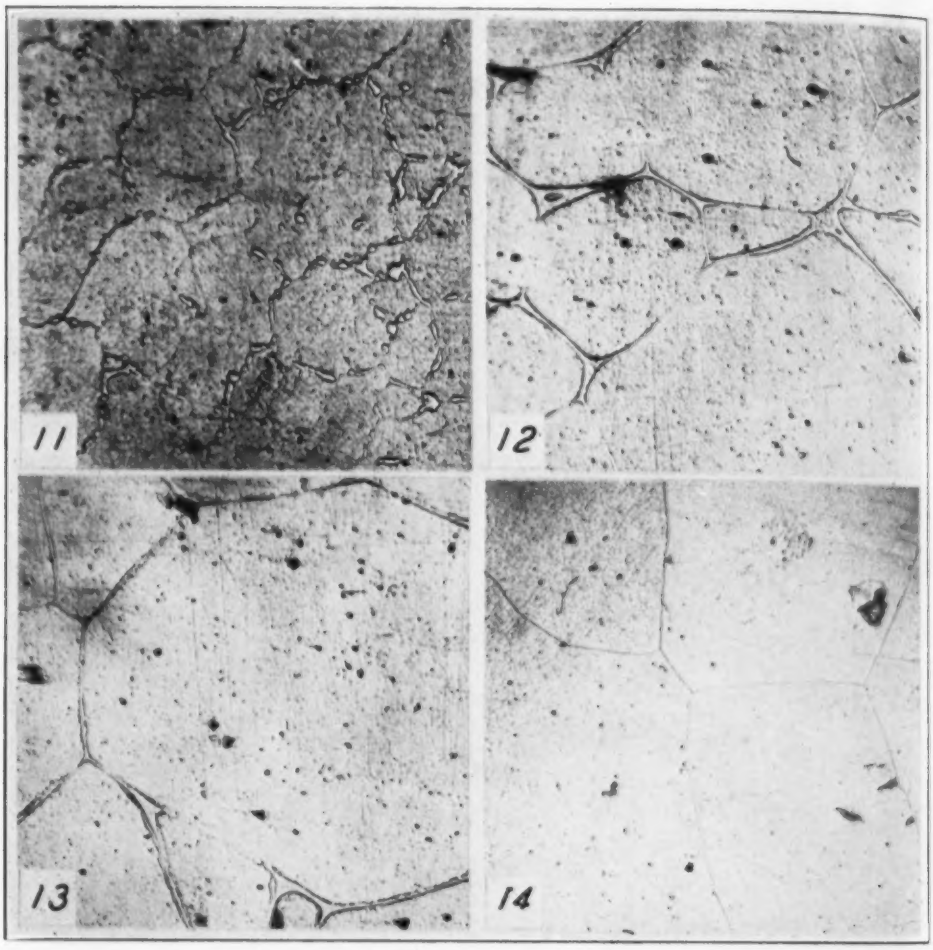


Fig. 11—Chromium 27.00 Per Cent, Carbon 0.19 Per Cent. Quenched after 2 Hours at 1250 Degrees Cent. (2280 Degrees Fahr.). Fig. 12—Chromium 28.81 Per Cent, Carbon 0.16 Per Cent. Quenched after 2 Hours at 1350 Degrees Cent. (2460 Degrees Fahr.). Fig. 13—Chromium 27.00 Per Cent, Carbon 0.19 Per Cent. Quenched after 2 Hours at 1350 Degrees Cent. (2460 Degrees Fahr.). Fig. 14—Chromium 27.85 Per Cent. Carbon 0.07 Per Cent. Quenched from 1350 Degrees Cent. Delta Iron Solution. All Specimens Etched with Aqua Regia in Glycerine. × 100.

point. Only occasionally in the vicinity of a large carbide particle of the annealed structure a small lake of austenite is produced at about 2190 degrees Fahr. (1200 degrees Cent.) which at higher temperatures, disappears . . . In alloys containing less than 0.25 per cent carbon it frequently occurs that no austenite can be found at all in the specimens regardless of the quenching temperature." More recently, Krivobok and Grossmann[4] in their comprehensive study of iron-chromium-carbon alloys found austenite in quenched

[4]Krivobok and Grossmann, "A Study of the Iron-Chromium-Carbon Constitutional Diagram," TRANSACTIONS, American Society for Steel Treating, July 1930, Vol. XVIII.

samples of 28 per cent chromium steel containing 0.32 per cent carbon. The dotted line in their diagram places the maximum carbon content for gamma free alloys at about 0.20 per cent.

In order to obtain further information on this point the writers melted in the high-frequency furnace three five pound heats of the following composition:

	No. 1 per cent	No. 2 per cent	No. 3 per cent
Carbon	0.15	0.19	0.27
Chromium	28.81	27.00	27.82
Silicon	0.45	0.34	0.34
Manganese	0.40	0.38	0.39

After the castings had been homogenized, specimens 1 by ½ by 3/16-inch were cut from the central portions of the castings. Pieces 3/16-inch thick were selected, instead of the thinner strips preferred by previous investigators, in order to minimize and to determine the extent of decarburization associated with the heating of this alloy to high temperatures. Preliminary work indicated that the treatment of thin strips led to chemical alterations of a serious nature. It is fortunate in this connection that the phase transformations in this alloy are so sluggish as to permit the use of reasonably thick specimens.

One specimen of each composition was quenched in water after heating for two hours at the following temperatures, 2100, 2280, 2370, 2460, 2570 and 2640 degrees Fahr. (1150, 1250, 1300, 1350, 1410 and 1450 degrees Cent.) requiring a total of eighteen specimens. Pieces from the three castings were heated together at each common temperature. In all cases, the steel was heated slowly to the desired temperature and then held at that temperature for the time specified. The heat treatments were carried on in a tightly sealed platinum-wound resistance furnace, the atmosphere of which approached as near a neutral atmosphere as may be conveniently realized.

For the preparation of the heat treated specimens suitable for chemical analysis and microscopic study, extreme care is necessary to insure the microscopic examination of a surface of known composition. It was observed that a film of oxide covered the specimens quenched from the lower temperatures; the specimens heated to the higher temperatures, however, were covered by a scale approximately 0.02-inch thick. The scale and decarburized under surface

(conclusive evidence of the serious decarburization was obtained by carbon determinations at various depths) were removed from all surfaces of the pieces. Material from one top face was removed until a plane was reached lying approximately half-way through the initial thickness. From that surface light chips were removed for a carbon determination; the machined surface, necessarily the most adjacent plane to that analyzed obtainable, was prepared for microscopic examination. In general, the samples for chemical analysis and structural studies contained carbon contents equal to those of the same specimens prior to the heat treatment.

In all of the specimens (0.15 to 0.27 per cent carbon) the presence of austenite in varying amounts was observed. In the case of the steels quenched from the lower temperatures, a large amount of the austenite was retained in the form of broad grain boundaries and spherical intergrain globules; in the specimens quenched from the higher temperatures a small amount of austenite was visible in the form of thinner grain boundaries and smaller intergrain globules. The pieces heated at 2640 degrees Fahr. (1450 degrees Cent.) showed evidences of liquefied metal. The presence of austenite is clearly shown in Figs. 10 to 13 which may be considered as typical of the structures observed.

Supplementary examinations of specimens subjected to the prolonged anneals referred to in the grain growth experiments disclosed a surface free from austenite. Although the alloy originally contained 0.26 per cent carbon, as a result of decarburization the plane examined had been reduced to 0.08 per cent carbon. An alloy containing 0.07 per cent carbon and 27.85 per cent chromium was then made by melting electrolytic iron and low carbon ferrochromium. This steel, upon quenching from temperatures ranging from 2190 to 2550 degrees Fahr. (1200 to 1400 degrees Cent.) contained no visible amounts of austenite. (Fig. 14.)

The experimental results of this study confirm the belief of Bain and others that carbon is soluble to an appreciable extent in delta iron, and since alpha iron at elevated temperatures becomes delta iron, it is evident that the solubility of carbon in chromiferous ferrite increases with increasing temperature. If the carbon content of the steel exceeds the solubility limit of carbon in delta iron the excess carbon prevents the complete suppression of the austenite and, therefore, manifests itself in the form of a solid solution of carbon in gamma iron. The solubility of carbon in delta iron (containing

28 per cent chromium) is apparently less than that reported by previous investigators. The writers believe that the limit is approximately 0.10 per cent carbon.

DISCUSSION

Written Discussion: By Sam Tour, vice-president, Lucius Pitkin, Inc., New York City.

The authors are to be congratulated on this very excellent piece of work. Their work has been on cast samples, which after annealing were given a 20 per cent reduction by cold working. The paper does not seem to give the reason for this. It is not clear whether the samples referred to in the first part of the paper dealing with the question of grain growth were heated in an oxidizing or a neutral atmosphere. The statements with regard to decarburizing would indicate that the samples were treated in an oxidizing atmosphere.

In connection with the heat treatment of tools made of an 18 per cent tungsten high speed steel, we have found grain growth at elevated temperatures is very much affected by the nature of the atmosphere in the furnace. For example, a piece of such steel ½ inch in diameter heated in still air at 2400 degrees Fahr. shows decided evidence of grain growth in 2½ minutes whereas a duplicate piece heated in a neutral or slightly reducing atmosphere to the same temperature and soaked four times as long, that is 10 minutes, does not show grain growth. In other words, in the case of 18 per cent tungsten high speed steel grain growth takes place in an oxidizing atmosphere but not in a true neutral or slightly reducing atmosphere. I have recently described some of these results in a brief article in the September 1930 issue of *Fuels and Furnaces*.

In studying this paper of Phillips and Baker, it occurred to me that possibly grain growth in high chromium-low carbon steel might be affected by furnace atmospheres.

A bar of ¾ inch diameter hot-rolled, annealed and pickled "Defiheat" rustless iron was obtained through the courtesy of the Rustless Iron Corp. of America. The mill reported the following analysis:

Cr	25.000%
C	.168%
Mn	.330%
P	.024%
S	.020%
Si	.750%

Seven pieces about one inch in length were cut from this bar and numbered from one to seven and treated as follows:

1. Not treated.
2. 1650 degrees Fahr.—4 hours Oxidizing atmosphere.
3. 1650 degrees Fahr.—4 hours Neutral atmosphere.
4. 2000 degrees Fahr.—4 hours Oxidizing atmosphere.
5. 2000 degrees Fahr.—4 hours Neutral atmosphere.
6. 2400 degrees Fahr.—4 hours Neutral atmosphere.
7. 2400 degrees Fahr.—4 hours Oxidizing atmosphere.

The furnace used for this work was a globar electric furnace as made

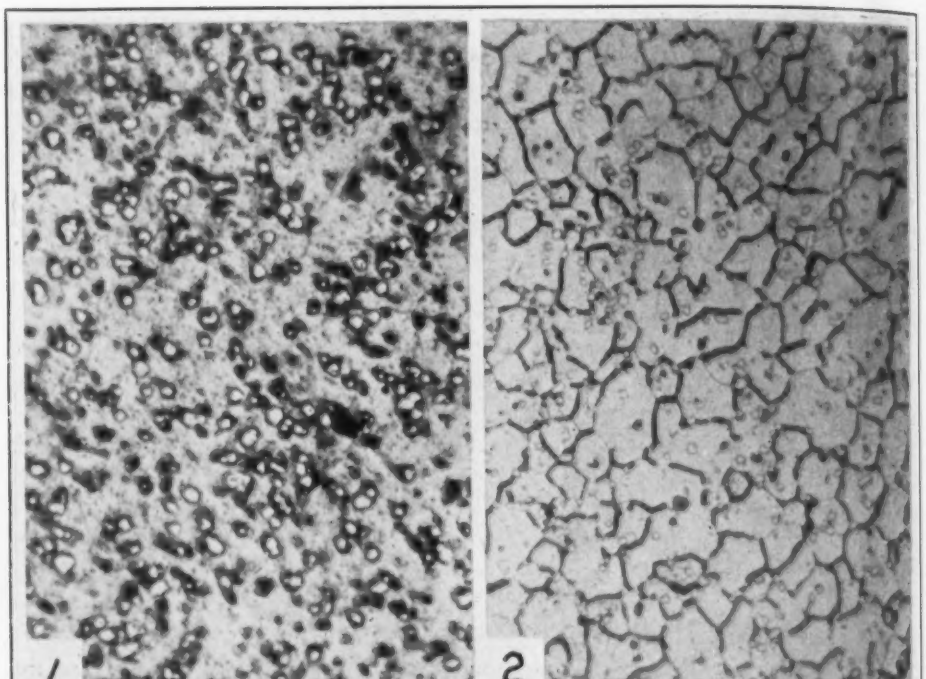


Fig. 1—Sample #1 Untreated. Fig. 2—Sample #2, 1650 Degrees Fahr., 4 Hours Oxidizing. Fig. 3 Sample #3, 1650 Degrees Fahr., 4 Hours Neutral. Note About Same Grain Size But Less Broadening of Grain Boundaries Than in the Oxidizing Treatment. Fig. 4—Sample #4, 2000 Degrees Fahr., 4 Hours Oxidizing. Note Large Number of Dark Spots on Apparent Oxides Within the Grains.

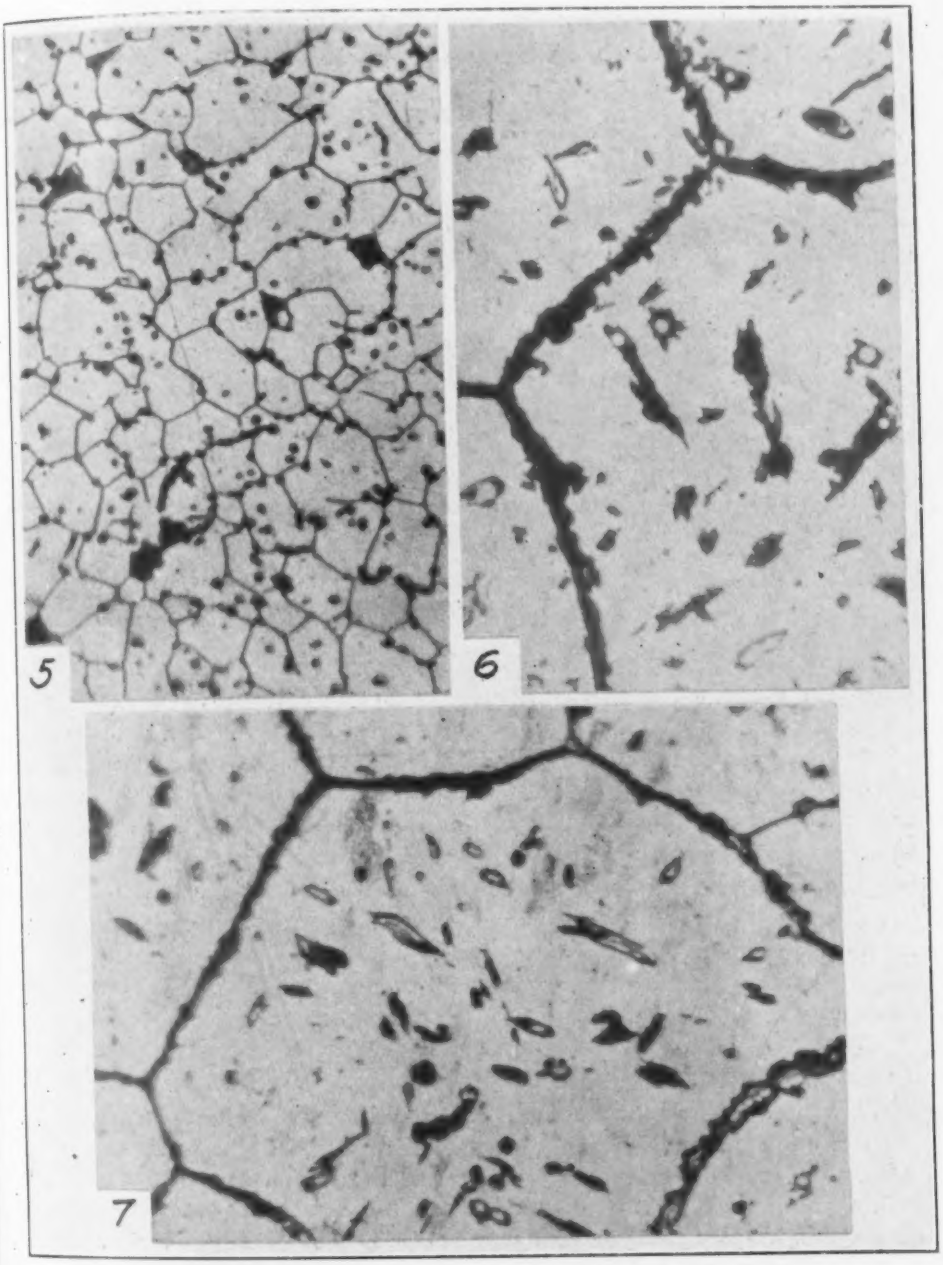


Fig. 5—Sample #5, 2000 Degrees Fahr., 4 Hours Neutral. Note Much Fewer and Much Smaller Spots Than in Oxidizing Treatment. Fig. 6—Sample #6, 2400 Degrees Fahr., 4 Hours Neutral. Fig. 7—Sample #7, 2400 Degrees Fahr., 4 Hours Oxidizing. Note Faint Gray Spots in Crystals. At Higher Magnification These Spots Show up Throughout all of the Grains in the Sample Treated in the Oxidizing Atmosphere Whereas They are not Evident in the Grains of the Sample Treated in the Neutral Atmosphere.

by C. I. Hayes, Inc. of Providence, R. I., and known as their "certain-curtain" furnace. Fig. A.

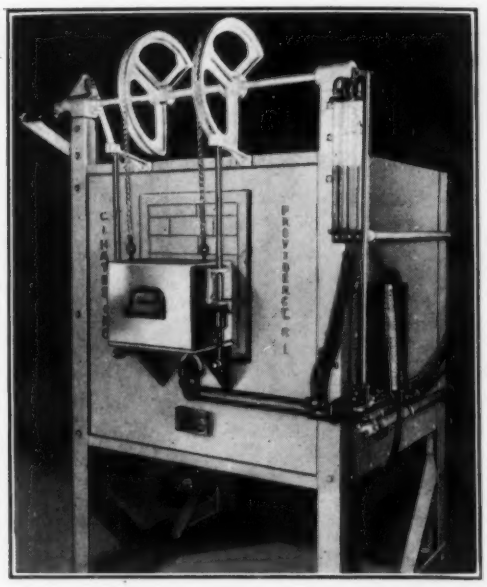


Fig. A

In this furnace carefully controlled proportions of air and gas are admitted into a small combustion space underneath the furnace hearth where they burn and the products of combustion rise through a slot at the front of the hearth and fill the heating chamber and act as a curtain to prevent in-leakage of air at the door. The ratio of gas to air is easily and accurately controlled by means of the two needle valves and manometers shown at the right of the illustration.

In these experiments, the oxidizing atmosphere was obtained by simply closing both the gas and air valves and the neutral atmosphere by proportioning one volume of gas to five of air.

All of these micrographs are at 100 diameters and show the structures as developed by etching with aqua regia in glycerine.

The results obtained would indicate that in this case grain growth is not greatly affected by furnace atmospheres. The actual grain growths obtained are comparable to those reported by Phillips and Baker.

Attention is called to the fact, however, that the difference in atmospheres has resulted in internal metallographic changes of the material within the grains. It is suggested that in the future experimenters on the effect of various heat treatments on microstructures take into account the question of furnace atmospheres.

Written Discussion: By Robert H. Aborn, member of the staff, United States Steel Corp., research laboratory, Kearny, N. J.

Thorough investigation of a series of alloys restricted to a small range of alloy content, such as has been described by Prof. Phillips and Mr. Baker, will continue to fill a real need in supplementing the pioneer work of Bain and the later comprehensive study by Krivobok and Grossmann on the iron-chromium-carbon system.

A word of caution with regard to procedure would seem advisable in view of the use of cast (unworked) structures by the authors for microscopic analysis of structural behaviors. Due to the marked atomic immobility of these alloys, complete chemical uniformity is extremely difficult to achieve except when the accelerating influence of deformation is applied. It is believed that the use of severely worked material as a basis for such investigations is more likely to yield thoroughly dependable results. Any appreciable lack of chemical homogeneity may, for example, result in the premature appearance of a new structural phase. Doubtless Prof. Phillips and Mr. Baker have in this case assured themselves of microscopic chemical homogeneity by comparison of the cast and annealed specimens with their own cold-worked grain growth specimens.

Authors' Closure

The question raised by Mr. Aborn regarding the possible lack of homogeneity attained by heat treating the cast alloys is well taken. The authors, however, were aware of the sluggishness of reaction in the high-chromium alloys and, therefore, made supplementary observation on hot-forged specimens and the moderately cold-worked pieces of the grain-growth series. From the confirmatory results obtained in this manner it is believed that equilibrium was attained in the original experiments.

Mr. Tour's remarks on the effect of atmosphere on grain growth are most interesting. Without question, in many cases, at the higher temperatures the furnace atmosphere plays an important part in grain growth. We know little about this matter, aside from the knowledge that marked changes in chemical concentration result from reaction between the gases and metal.

The heat treatments of the grain growth series were carried on in a platinum-wound resistance furnace sealed at both ends. The atmosphere was, therefore, still air. We are pleased to learn that Mr. Tour's grain growth results in both neutral and oxidizing atmospheres are substantially the same as reported in our paper.

DATA ON MANGANESE STRUCTURAL STEELS WITH CHROMIUM ADDITIONS

By A. B. Kinzel and W. B. Miller

Abstract

This paper presents the results of a laboratory study on a series of chromium-manganese steels ranging from 0.08 to 0.50 per cent carbon and 0.6 to 1.8 per cent manganese, with chromium additions from 0.3 to 1.1 per cent. The beneficial effect of chromium is evidenced by the increase of ultimate strength without proportional decrease in ductility. The practical application of such a steel is demonstrated by the construction of a welded pressure vessel and its subsequent test to destruction. The possibility of as rolled or normalized structural steels with satisfactory ductility and higher strength than the present manganese steel by the addition of small percentages of chromium to these steels is indicated.

Foreword

IN the past few years the application of steels with 1 to 2 per cent manganese content for structural purposes has increased at a very rapid rate, and these steels may now be considered as being in common use. Silicon-manganese steels, both the 0.40 per cent silicon and the 1 per cent silicon types, have also been applied in industry, and very recently silicon-manganese-chromium steel has come into use, some 20,000 tons of this steel having been manufactured in the past year. The addition of chromium in relatively small amounts to the manganese structural steels is not new, but there has been very little published of a systematic nature regarding the properties of chromium-manganese steels in the as rolled or normalized condition. As steels for structural applications are generally limited in heat treatment to simply cooling from rolling or normalizing, this study has been similarly restricted. The following data indicate the nature of the changes in physical properties produced by these additions.

A paper presented at the semi-annual meeting of the American Society for Steel Treating in New York City, February 7 and 8, 1930. The authors, members of the society, are associated with the Union Carbide and Carbon Research Laboratories, Inc., Long Island City. Manuscript received December 26, 1929.

Table I

Analysis of Manganese-Chromium Steels

Heat	Per Cent Manganese	Carbon	Chromium	Silicon
1	0.67	0.07	0.33	0.11
2	0.82	0.07	0.38	0.13
3	0.91	0.09	0.41	0.07
4	0.93	0.08	0.38	0.08
5	1.13	0.10	0.70	0.10
6	1.16	0.13	0.60	0.12
7	1.22	0.08	0.49	0.12
8	1.46	0.07	0.82	0.13
9	1.70	0.09	1.05	0.17
10	0.63	0.26	0.42	0.12
11	0.69	0.26	0.63	0.14
12	0.89	0.22	1.13	0.10
13	0.90	0.25	0.45	0.09
14	1.11	0.17	1.37	0.17
15	0.78	0.31	1.03	0.21
16	1.17	0.37	1.04	0.21
17	1.35	0.44	1.12	0.34
18	1.37	0.50	0.31	0.22
19	1.73	0.44	0.64	0.14
20	1.75	0.43	0.40	0.34

Sulphur and phosphorus low in all cases.

Composition and Production

The chemical composition of the heats studied is given in Table I. These heats were made in a 6-pound induction furnace and forged and rolled to 1-inch rounds. In addition, a number of heats of manganese steel without chromium additions have been made in the same way. While some of these manganese steel heats were made particularly in connection with the present investigation, a great many have been made prior to this. Moreover, as the properties of these manganese steels are well known, it is not the purpose of this paper to discuss them, except as they are needed for comparison. Accordingly, these physical properties are plotted in the curves shown in Figs. 1 to 4 without further comment. The writers appreciate that the results obtained on steel manufactured in this way are not strictly comparable to steels made in the mill, but they are strictly comparable among themselves. Moreover, general experience with the manganese steels, as well as the specific experience with the manganese steel with chromium addition, later described herein, indicates that a very reasonable agreement exists between steels made with proper laboratory practice and those made with ordinary mill practice.

Table II

Physical Properties of Normalized Manganese-Chromium Steels

Heat	Per Cent Mn	Per Cent C	Per Cent Cr	Yield Point	Ultimate Strength	Elong. Per Cent 2 inches	Red. of Area Per Cent	Izod	Brinell Number
1	0.67	0.07	0.33	42,000	59,000	38	71	87	104
2	0.82	0.07	0.38	43,000	60,000	37	69	90	107
3	0.91	0.09	0.41	42,000	61,000	36	67	87	107
4	0.93	0.08	0.38	42,000	60,000	39	71	86	110
5	1.13	0.10	0.70	43,000	67,000	36	71	84	121
6	1.16	0.13	0.60	45,000	74,000	38	71	89	129
7	1.22	0.08	0.49	46,000	66,000	37	73	86	117
8	1.46	0.07	0.82	48,000	71,000	32	60	74	125
9	1.70	0.09	1.05	52,000	90,000	26	52	37	171
10	0.63	0.26	0.42	54,000	85,000	29	56	55	143
11	0.69	0.26	0.63	54,000	87,000	28	62	41	202
12	0.89	0.22	1.13	57,000	95,000	25	62	41	187
13	0.90	0.25	0.45	54,000	85,000	30	56	56	146
14	1.11	0.17	1.37	58,000	96,000	24	56	35	170
15	0.78	0.31	1.03	66,000	109,000	23	59	36	202
16	1.17	0.37	1.04	107,000	148,000	5	8	34	146
17	1.35	0.44	1.12	92,000	162,000	3	4	..	...
18	1.37	0.50	0.31	75,000	127,000	15	27	7	241
19	1.73	0.44	0.64	114,000	141,000	3	3	4	293
20	1.75	0.43	0.40	94,000	138,000	12	25	.	...

Test Specimens

The physical properties of the normalized manganese steels with chromium additions are given in Table II. The normalizing consisted of heating 1-inch rounds 6 inches long, at 1650 degrees Fahr. (900 degrees Cent.) for 30 minutes after the outside attained temperature, and cooling in still air, with the pieces standing on end. Standard 0.505-inch tensile test specimens and the usual 0.394-inch notched Izod specimens were prepared from these 1-inch rounds. In addition, a similar series of tensile and impact specimens was prepared from 1-inch rounds, as forged and rolled. Physical properties of the material, as forged and rolled, are given in Table III.

Test Results

Fig. 1 shows the ultimate strength obtained on specimens treated as described above with varying manganese and carbon and no chromium, the curved lines representing various carbon contents. Fig. 5 shows the same curves with vertical lines superimposed on these curves, to show the improvement in ultimate strength obtained by the chromium addition. The solid dot at the foot of each vertical line represents the ultimate strength obtainable with each of these steels

without chromium, and the circle at the upper end of the vertical line shows the ultimate strength obtained with the chromium addition. The small figures near each line show the amount of the chromium addition. It will be seen that this curve may also be used as a nomographic chart. Thus, the circle at the upper end of the line represents the alloy of the same manganese content with no chromium, but with the carbon content increased so that the steel has the ultimate strength indicated. This ultimate strength is the same

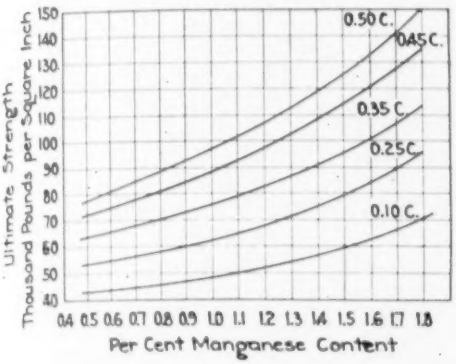


Fig. 1—Ultimate Strength of Normalized Metals with Varying Carbon and Manganese Content.

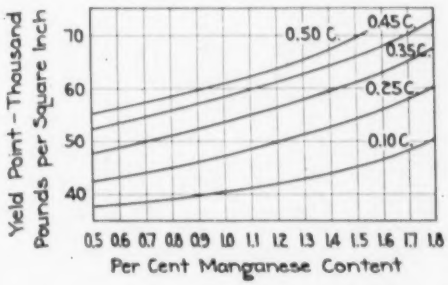


Fig. 2—Yield Point of Normalized Manganese-Carbon Steels.

as the ultimate strength of the lower carbon steel with the chromium addition. Likewise, the horizontal line through the upper circle represents the constant ultimate strength, so that by following this line to the right until it intersects the same carbon curve which passed through the lower dot, the manganese content of the steel having the same ultimate strength and the same carbon content as the steel with the chromium addition, is obtained. As a specific instance, the first chromium-manganese alloy steel plotted on the lower part of the diagram has approximately 60,000 pounds per square inch ultimate strength. This was obtained by the addition of 0.33 per cent chromium. Without this chromium but with the same carbon and manganese, the steel has an ultimate strength of 43,000 pounds per square inch. Without chromium and with the same manganese, but with the carbon increased to 0.28 per cent, the steel also has 60,000 pounds per square inch ultimate strength. Again, by following the horizontal through the circle at 60,000 pounds per square inch until it intersects the 0.08 per cent carbon curve, it is shown that with 1.6 per cent manganese, 0.08 per cent carbon and no chromium, 60,000 pounds per square inch ultimate

Table III
Physical Properties of Manganese-Chromium Steels as Forged and Rolled

Heat	Mn	Per Cent C	Cr	Yield Point	Ultimate Strength	Per Cent Elong. in 2 Inches	Red. of Area Per Cent
1	0.67	0.07	0.33	45,000	59,000	36	66
2	0.82	0.07	0.38	43,000	51,000	41	75
3	0.91	0.09	0.41	41,000	52,000	39	71
4	0.93	0.08	0.38	42,000	61,000	39	70
5	1.13	0.10	0.70	46,000	56,000	31	68
6	1.16	0.13	0.60	47,000	74,000	32	63
7	1.22	0.08	0.49	45,000	67,000	38	63
8	1.46	0.07	0.82	43,000	75,000	28	60
9	1.70	0.09	1.05	50,000	89,000	23	44
10	0.63	0.26	0.42	54,000	84,000	25	37
11	0.69	0.26	0.63	66,000	105,000	22	55
12	0.89	0.22	1.13	69,000	100,000	14	49
13	0.90	0.25	0.45	58,000	86,000	29	54
14	1.11	0.17	1.37	59,000	95,000	24	47
15	0.78	0.31	1.03	66,000	105,000	22	55
16	1.17	0.37	1.04	116,000	127,000	1	2
17	1.35	0.44	1.12	.....	.....	..	..
18	1.37	0.50	0.31	80,000	123,000	11	..
19	1.73	0.44	0.64	.....	.....	..	..
20	1.75	0.43	0.40	82,000	123,000	13	35

strength again is achieved. It is a quite obvious and well-known fact that ultimate strength may be obtained in various ways. The difference in the properties of the steels, then, is found largely in the ductility.

Discussion of Test Results

The steels are arranged in the various tensile groups in Table IV, and the ductility of the steels in each group is listed. The curve, Fig. 7, shows the improvement in ductility obtained with chromium additions. The manganese steel in each group is chosen so as to have approximately the same carbon content as the manganese-chromium steels. This shows that the ductility of manganese steels containing chromium is greater than that of the plain manganese steels of the same carbon content and the same ultimate strength. This increase in ductility is particularly noticeable in the reduction of area. Moreover, as the ultimate strength in question is increased, the difference in ductility is increased, so that on reaching steels of the order of 110,000 and 125,000 pounds per square inch with the same carbon content a very real gain in ductility is achieved by the chromium addition. As to the quantity of the chromium addition, it will be noted from the diagram, Fig. 5, that the higher the carbon content the more effective a given amount of chromium, and the higher the

manganese content the less effective a given amount of chromium.

Another way of stating the results is that for a given ductility the ultimate strength of the chromium-manganese steel is greater than that of the plain manganese steels. One of the broad generalizations which has been made frequently regarding alloy steels is that the lower the carbon content for a given ultimate strength, said

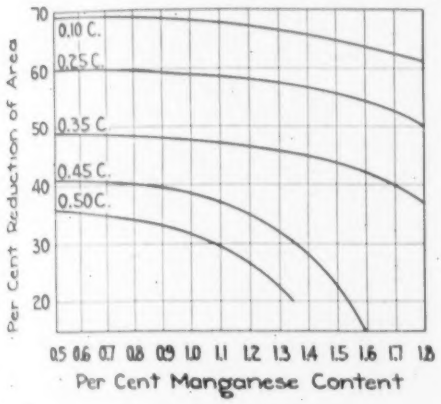


Fig. 3—Elongation in 2 Inches on Normalized Manganese-Carbon Steels.

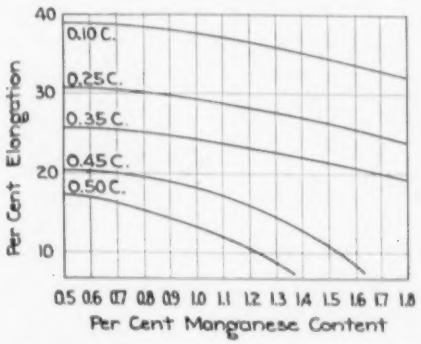


Fig. 4—Reduction of Area of Normalized Manganese-Carbon Steels.

ultimate strength being achieved by means of suitable alloy additions, the greater the ductility. In view of this, the comparison made above is based on manganese steels of the some carbon content. Were manganese steels of higher carbon content and lower manganese content chosen for comparison, the difference in ductility would be even more striking. The above statements may be summarized in the statement that chromium additions to manganese steels increase the ultimate strength without proportionate loss of ductility in the normalized state.

It is of interest to consider the analysis of a chromium-manganese steel which would meet the minimum 90,000 pounds per square inch ultimate strength specified for the 0.30 per cent carbon, 1.60 per cent manganese structural steel now in use. A study of the data here given indicates that this would result from a 0.30 per cent carbon, 1.25 per cent manganese, 0.30 per cent chromium steel, or from a 0.30 per cent carbon, 1.40 per cent manganese, 0.20 per cent chromium steel, and that the ductility would be greater than that of the plain manganese steel. Likewise, this ultimate strength would result from a 0.25 per cent carbon, 1.25 per cent manganese, 0.35 per cent chromium steel, or from a 0.25 per cent carbon, 1.40 per

Table IV

Ductility of Manganese-Chromium Steels Compared to Manganese Steels of the Same Carbon Content and the Same Ultimate Strength

Tensile Group Pounds Per Square Inch	Steel	Per Cent Elongation in 2 Inches	Per Cent Red. of Area
	Steels 1, 2, 3, 4	38	69
60,000	Mn 1.6%—C .08%	39	65
	Steels 5, 6, 7	37	72
70,000	Mn 1.8%—C .10%	32	62
	Steels 10, 11, 12, 13, 14	29	60
90,000	Mn 1.7%—C .25%	25	52
110,000	Steel 15	23	59
	Mn 1.75%—C .35%	20	39
125,000	Steel 18	15	27
	Mn 1.60%—C .45%	8	15

cent manganese, 0.25 per cent chromium steel, with even greater improvement in ductility. Moreover, the maximum limits of manganese and carbon, consistent with satisfactory ductility for structural purposes in the as rolled or normalized states, have been almost reached in current manganese steels, and the addition of chromium opens the way to higher strength structural steels with satisfactory ductility in the as rolled or normalized condition.

Little can be said regarding the properties in the as forged and rolled condition, except that with the small scale practice used in the laboratory a very reasonable check between the two exists, particularly with respect to yield point and ultimate strength. However, the ductility of the as forged and rolled steels is in general distinctly lower than that of the steels as normalized, particularly as indicated by reduction of area, and most noticeably in the higher ultimate strength steels. As the majority of the Izod values are obtained from material which bent rather than fractured on test, no quantitative comparison may be made. Only future experience will tell whether the same relation of properties of the steels as rolled to the properties of the steels as normalized will result with the material rolled according to standard mill practice.

Mill Production of a Manganese-Chromium Steel Pressure Vessel

A special heat of chromium-manganese steel for the purpose of a weld test has been made at a well-known steel mill for the Union

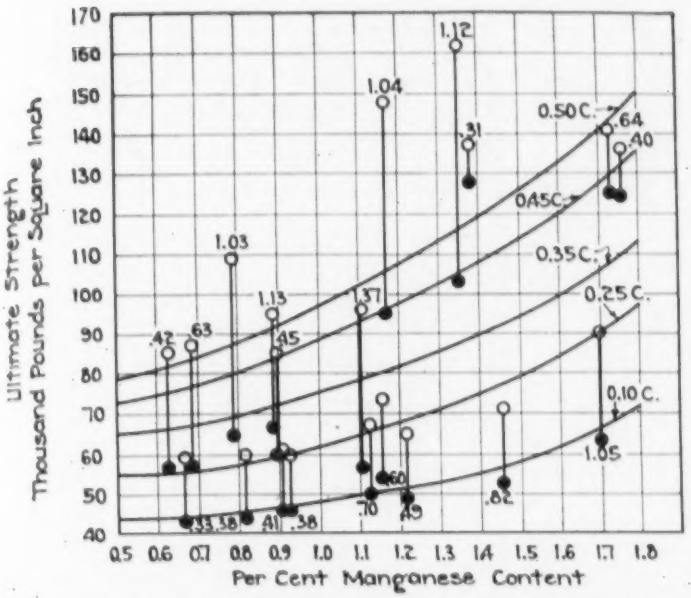


Fig. 5—Chart Showing Improvement in Ultimate Strength of Normalized Manganese Steels, Due to Chromium Addition.

Carbide and Carbon Research Laboratories, and is illustrative of the correlation of the work done in the laboratories with mill results. This heat analyzed carbon 0.28 per cent, manganese 0.90 per cent, phosphorus 0.028 per cent, sulphur 0.039 per cent, silicon 0.33 per cent, nickel nil, chromium 0.31 per cent. It was made in a 6-ton electric furnace and rolled according to standard practice to $\frac{3}{8}$-inch plate. No difficulties in furnacing or rolling were experienced. The plate was formed to the same sized tank used by the American Bureau of Welding at the United States Bureau of Standards in 1922, 24 inches in diameter, but deviated from this tank in that the cylindrical section was 6 feet long and that long skirt elliptical heads were welded on each end. Thus, the over-all length was 7 feet 9 inches, instead of 6 feet. The design and proportions of the tank are shown in Fig. 6. This tank was welded under procedure control with a manganese-molybdenum high strength welding rod, known to the trade as No. 22. The steel showed excellent welding qualities. After welding, the tank was heated for one hour at 1650 degrees Fahr. (900 degrees Cent.), cooled to a black heat in the furnace and then cooled in air.

Testing of the Manganese-Chromium Steel Pressure Vessel

This tank was subsequently tested to destruction at the Union

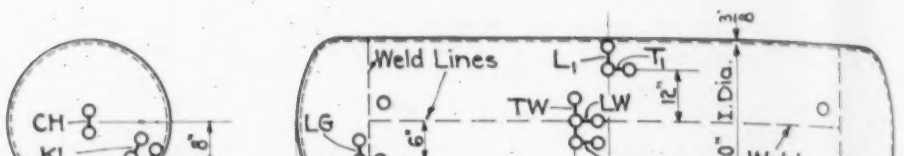


Fig. 6—Experimental Tank Showing Location of Strain Gage Readings.

Table V
Strain Gage Readings at Reference Points on Tank Shell and Head

Pressure Lbs. Per Square Inch	T_1	L_1	CH	KL_1	KL_2	KR	TG	LG	TW	LW	L_1W	Girth Measure
0	95	90	101	116.7	73.7	111	115.5	141.5	106	117	114.7	
500	98	90	108	113	70.5	114	116.9	145	129	126	116.5	
1000	104.5	90.6	113.3	109	64.5	118	118.8	149.5	162.5	127.5	118	
0	95	88.3	102	116	72	112	115	141	144	124	115	6'6"
1800	199.5	92	196	101	50	127.5	116.5	162	238	127	117	6'6¾"
2450						Rupture						6'9¾"
After Rupture	....	....	306	40	9	186	140	305	....	141	122	

Carbide and Carbon Research Laboratories. It failed at a hydrostatic pressure of 2450 pounds per square inch, or 78,500 pounds per square inch fiber stress. Readings taken before rupture showed that the circumference of the center of the tank had increased 3¾ inches. Strain gage readings at reference points on the tank and shell shown in the accompanying sketch, Fig. 6, show the nature of the deformations. It should be noted that excellent ductility was evident and that the failure occurred at a relatively high fiber stress. Failure took place outside of the weld at all points, and at no place did the fracture run into the weld, although at some points it ran very close to it and parallel to it. Subsequent to the testing, specimens were cut from the tank, and plate material was tested in both longitudinal and transverse sections. In addition, samples were taken across the longitudinal weld and tested in the same way. The results of these tests are shown in Table VI. Good correlation exists between properties of the steel made in the mill with the properties which we should have expected from the laboratory results. This is particularly true in view of the difference in heat treatment and dimensions of test specimens.

Table VI

Physical Properties of Plate Taken from Pressure Vessel of Manganese-Chromium Steel

Standard Flat Specimen	Yield Point	Ultimate Strength	Per Cent of Elongation
Plate material, longitudinal	66,500	92,000	..
Plate material, longitudinal	65,200	91,100	..
Plate material, longitudinal	.....	.....	27
Plate material, transverse	76,700	95,400	..
Plate material, transverse	76,100	94,900	..
Plate material, transverse	.....	.....	27
Plate with welded seam, transverse	77,500	97,600	..
Plate with welded seam, transverse	Bend Elongation		25
Longitudinal welded seam with reinforcement ground off	Bend Elongation		22

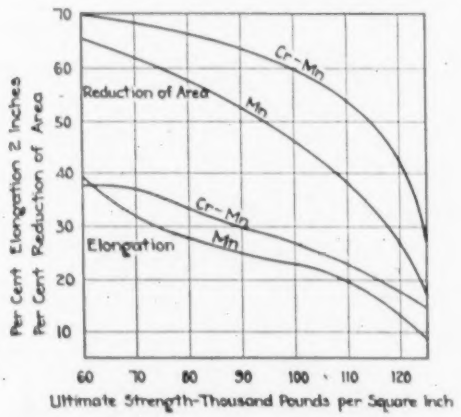


Fig. 7—Elongation and Reduction of Area of Manganese and Chromium-Manganese Steels of the Same Carbon Content as a Function of the Ultimate Strength.

Silicon-Manganese-Chromium Steels[1]

A discussion of this subject would be far from complete were the matter of the same steels with higher silicon content not mentioned. It will be noted that the silicon content in the steels previously referred to does not exceed 0.35 per cent, and in most cases is appreciably lower. The physical properties of silicon-manganese-chromium steels are compared to those of manganese-chromium steels in Table VII. The mode of manufacture and heat treatment of the series of steels are identical. Thus, they may be directly compared. The beneficial effect of the silicon in this alloy steel is very evident. However, the alloy content of these steels is somewhat greater than that of the manganese-chromium steels, and for this reason, if for no

[1]A. B. Kinzel, "Silicon-Manganese Steels with Chromium Additions for Engineering Applications," Transactions, American Society for Steel Treating, Vol. 14, Dec., 1928, p. 866.

Table VII

Comparison of Physical Properties of Manganese-Chromium and Silicon-Manganese-Chromium Steels

Steel	Yield Point Pounds Per Square Inch	Ultimate Strength Pounds Per Square Inch	Per Cent Elongation in 2 Inches	Per Cent Reduction of Area
Si-Mn-Cr	72,000	111,000	25	60
Steel 10	54,000	85,000	26	52
11	54,000	87,000	23	62
12	57,000	95,000	25	62
14	58,000	96,000	24	56
Si-Mn-Cr	79,000	126,000	21	59
18	75,000	127,000	15	27

other, the steels may well be placed in a separate category, leaving a particular field for the manganese-chromium steels without silicon.

Conclusions

The writers realize that conclusions drawn from the data here presented pertain to small scale operation and that these conclusions may require modifications if applied to steel produced in the mill. However, their past experience, particularly with the silicon-manganese-chromium steels, indicates that the general nature of the alloy effect holds as well in mill production as it does in laboratory heats. Conclusions which may be drawn from the data here presented follow:

1. The addition of small percentages of chromium to manganese steel increases the ultimate strength without proportionate decrease in ductility.

2. The addition of small percentages of chromium to manganese steel gives greater ductility for the same ultimate strength and the same carbon content.

3. The effect of the chromium increases with the carbon content, and decreases with the manganese content of the steel in question.

4. The properties of the manganese-chromium steels are such as to render them decidedly applicable in the general structural field.

5. The practical application of such a steel has been demonstrated by the construction of a welded pressure vessel and its subsequent test to destruction.

6. The possibility of as rolled or normalized structural steels with satisfactory ductility and higher strength than the present man-

ganese steels may be realized by the addition of small percentages of chromium to these steels.

Acknowledgment

The writers wish to express their appreciation of the stimulating interest shown by Messrs. F. M. Becket and J. H. Critchett, president and vice president of the Union Carbide and Carbon Research Laboratories. They also wish to thank their colleagues at the laboratory, particularly Messrs. C. O. Burgess and C. H. Kluge, for work on the experimental heats, and C. W. Obert for advice and assistance in testing the tank. Strain gage measurements in this work were taken by T. W. Green, of the Linde Air Products Co., to whom we are also indebted.

DISCUSSION

A. V. DeForest[1]: About a year and a half ago the Spang Chalfant Co. was faced with the necessity of improving a straight carbon steel analysis under several different conditions of heat treatment. What we desired was improvement which would reflect both in the "as rolled" properties and in the normalized properties of seamless tubing. If possible, the use of the same analysis would be desirable in a quenched and drawn condition for other uses. We experimented at length with the subject of Mr. Kinzel's former paper, the silico-manganese-chromium steel, and got very excellent results from that analysis. The combination is flexible and almost any mechanical properties can be obtained from that type of alloy. For less rigorous specification, we have been experimenting with the manganese-chromium steel, particularly the manganese-chromium steel as in this paper. It is largely by the presentation of papers such as this, which tabulate results in an orderly form, that the industries can quickly make use of new developments in the alloy steels. I know that we at Spang Chalfant have been helped enormously by the work that the Carbide Co., has been willing to publish on these structural steels.

R. H. Rowley[2]: In regard to Mr. Kinzel's statement that we hope to see steel of approximately 110,000 pounds to 120,000 pounds per square inch tensile strength, I wish to state that steel of this strength is now being furnished to users throughout the oil field of the middle west and Pacific coast regions. This type of steel is being fabricated into casing and drill pipe by three companies and they are meeting this strength without the aid of chromium or heat treatment.

This steel is approximately 0.40 to 0.50 per cent carbon and about 1.22 per cent manganese and the tube makers guarantee minimum tensile strengths

[1]Member A. S. S. T., consulting metallurgist, Spang Chalfant Co., Pittsburgh.

[2]Member A. S. S. T., chief inspector, Monessen Works, Pittsburgh Steel Products Co., Pittsburgh.

of 95,000, 115,000 and 125,000 pounds per square inch respectively on grades 1, 2 and 3.

A. B. KINZEL: The figures quoted by Mr. Rowley are certainly splendid. However, it must be remembered that these are obtained on comparatively thin-walled tubing, and that the figures quoted in this paper are obtained on one-inch rounds. Working with a steel of the same carbon range as Mr. Rowley's, the addition of chromium raised the ultimate strength in the as rolled condition to 148,000 pounds per square inch with satisfactory ductility.

R. H. ROWLEY: I think the addition of chromium is a benefit to the steel and will increase its tensile strength without a loss of ductility and I attribute the greater strength of the tubing, over the 1 inch solid bar to the fact that the tube has a greater area exposed to the cooling effect of the air, due to the fact that it is hollow and cools both inside and outside at the same time.

ALEXANDER GOBUS[3]: Sometime ago I read of a tank which was tested somewhat as yours was tested, and on bursting it broke into 9 pieces. That is taken by some as a measure of the ductility. I would like to know if you agree with that and also into how many pieces your tank broke.

A. B. KINZEL: The tank in question did not fragment at all. It split open and remained in one piece. If this is a measure of ductility, the conclusion is obvious. Moreover, it probably is a rough measure of the true toughness, but a purely qualitative one. The form of the tank has much to do with the matter of fragmentation. Was your tank spherical or cylindrical?

ALEXANDER GOBUS: It was ball-shaped.

A. B. KINZEL: This tank was cylindrical, and it is possible that there is very litle correlation between the two.

W. J. MERTEN[4]: I want to ask Mr. Kinzel what fundamental reasoning made him add chromium. The trouble with manganese steels of the 1.5 per cent and over manganese content was particularly evident in larger sections, above 4 inches in diameter. We found that the larger sections were particularly sensitive to thermal treatment. Only with accurate control of temperatures were we able to produce a grain structure that was sufficiently fine to employ this material in shafting. We had no end of trouble with the ordinary 0.35 to 0.45 per cent carbon, 1.5 per cent manganese steels in still larger sections. I am wondering whether the refinements of grain due to the chromium additions and the less sensitive condition of steel to thermal treatments because of the presence of chromium, was not the fundamental reason for this development.

A. B. KINZEL: The hardening action and grain refinement of the chromium is undoubtly one factor. However, there are others. For example, with chromium the tendency for ingot segregation seems to be greatly reduced. This, we believe, is the most important reason. It probably accounts for the splendid commercial success obtained with these steels and the silicon-manganese-chromium steels.

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[3]Member A. S. S. T., research department, American Car and Foundry Co., New York City.

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[4]Member A. S. S. T., metallurgical engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh.

W. R. K. Scott[5]: I would like to ask Mr. Kinzel what are the manufacturing difficulties with carbon steels when the manganese is above 1.8 per cent.

A. B. Kinzel: Most steel manufacturers state that there are no difficulties with higher manganese steels. This is probably true with correct practice. However, some difficulties may be encountered and are exhibited by lessening the ductility of the material. This may probably be traced back to the ingot.

Chairman E. C. Bain[6]: Approximately, what is the elongation and reduction of area of the 150,000 pound per square inch steel that you made with high carbon chromium?

A. B. Kinzel: The elongation was 22 per cent, and the reduction of area 40 per cent, both taken on a rectangular 0.4″ x 1¼″ section, so that with 0.505-inch rounds even better figures would have been obained.

W. R. K. Scott: With the chromium additions to the steel of the carbon and manganese referred to, does it have a tendency to decrease the machinability for the same ultimate strength?

A. B. Kinzel: That question can only be answered by considerable shop experience. In general, machining is easier for the same ultimate strength and ductility.

Alexander Gobus: I forgot to ask whether that tank was heat treated in any way after welding.

Mr. Miller[7]: The dimension of the tank was three-eighths inch thick, shell six feet long by 2 ft. dia. and was rolled up and formed from the "as rolled" condition. There was some spring in it for it was finished at a relatively low finishing temperature. After welding the tank was normalized at 900 degrees Cent. for an hour in a very large furnace and had to cool down to almost a black heat in the furnace because we had no means of getting into the large hot furnace. As soon as it was black we were able to take it out and let it cool in still air. That was the only heat treatment the tank received.

O. W. Ellis[8]: Could Mr. Kinzel give us some information as to the method he adopted in making these alloy steels.

A. B. Kinzel: Both the carbon-manganese and the carbon-chromium-manganese steels here mentioned were made in the induction furnace. A low carbon melt was deoxidized with silicon and manganese, and the main manganese addition subsequently made. The chromium addition was last. The heat was allowed to come to proper temperature after the chromium addition, before pouring. The average time of holding after the chromium addition was three to five minutes. Six-pound heats were used. These are justified by our past experience showing that a certain correlation between these heats and steel made in the mill does exist.

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[5]Member A. S. S. T., metallurgist, Jones and Laughlin Steel Co., Aliquippa, Pa.

[6]Member A. S. S. T., research staff of U. S. Steel Corporation, Kearny, N. J.

[7]Member Union Carbide and Carbon Research Laboratories staff, Long Island City, N. Y.

[8]Member A. S. S. T., director of metallurgical research, Ontario Research Foundation, Toronto, Canada.

CHAIRMAN E. C. BAIN: I wonder if you would tell us the approximate chromium equivalent with constant carbon in the vicinity of 1.25 manganese. That is to say, how much manganese can we dispense with and still maintain the same ultimate strength by the addition of chromium.

A. B. KINZEL: The chromium-manganese equivalent varies with the carbon content. Broadly speaking, approximately two-thirds of a unit of chromium is equivalent to one of manganese.

CHAIRMAN E. C. BAIN: That is important from the cost standpoint.

A. B. KINZEL: From the cost standpoint, it should be remembered that we are dealing with alloy steels in which the alloy addition cost is extremely low. Silicon, manganese and chromium are the least costly alloy additions which can be made to steel. This is one reason why these steels have been so successful in industry.

W. J. MERTEN: I would like to ask Mr. Kinzel what his opinion would be in bringing that composition into locomotive castings.

A. B. KINZEL: Mr. Hamilton has used these steels in castings, and I shall ask him to reply.

MR. HAMILTON[9]: I might say that our company (American Steel Foundries) has made quite a good many large sized castings from this composition of manganese and chromium where the manganese is held at about 1.45 and the chromium between 60 and 70 and carbon between 35 and 45. We have had to meet specifications of 110,000 tensile; 65,000 tensile yield with 18 elongation and 24 reduction. We have been able to do that without encountering any difficulty in casting problems other than we would with either the straight manganese or the straight carbon steels. We do not believe that it is any more difficult to cast this type of steel than it is to cast the straight carbon. The matter of heat treatment of course has to be followed very closely to get the results that are required.

W. J. MERTEN: I would like to ask Mr. Hamilton if those tests are on the coupon type of bar about one and a half, two sections about 4 and 5 inches long.

MR. HAMILTON: Those results are based on the coupon test, of the regular size attached to the castings.

W. J. MERTEN: That is naturally the opinion that I have of the tests that you are giving us.

MR. HAMILTON: Yes, and this factor should be known. It is a very important consideration.

S. MARMOROFF: I would like to ask Mr. Kinzel whether this material can be hammer-welded.

A. B. KINZEL: It will probably be possible to hammer-weld these steels, although we have no experience with them in this connection.

W. J. MERTEN: I wonder if he has any impact values of those steels in the impact condition.

A. B. KINZEL: The steels in question treated to approximately 140,000 pounds per square inch have impact values varying from 14 to 22 pounds.

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[9]Member A. S. S. T., research director, American Steel Foundries Co., Indiana Harbor, East Chicago, Ind.

CORROSION AND HEAT RESISTANT NICKEL-COPPER-CHROMIUM CAST IRON

By J. S. Vanick and P. D. Merica

Abstract

These investigations, in the laboratory, foundry and field, have now been under way for some three or four years and it has been possible to survey in some detail the various characteristics of these cast irons through a wide range of chemical composition as well as to decide, as a result of these tests, upon a composition range most suited for general service.

Such a material, corresponding to the composition given, has now been in rather varied industrial service for periods up to about three years, and it has within that period been possible to gain a clearer picture of its practical serviceability and value.

Field service tests under a variety of conditions have confirmed in general the results of laboratory tests in establishing a greatly increased service life for this material as compared with plain iron, and a life comparable in many cases with bronze. It has further been demonstrated that this material, except for being considerably more corrosion and heat resistant than plain iron, is in general quite similar to it in characteristics and in fabricability. Thus it can be produced in the ordinary cupola, and can be molded, cast, and handled in foundry and machine shop under conditions similar to those in practice for plain gray iron. It may be readily cast, machined, and welded, although modifications of cast iron practice are in some cases necessary. In short, it is a material which may be produced in any well organized iron foundry and at a reasonably low cost.

SEVERAL years ago, during the earlier experimental work on nickel cast iron,[1] a preliminary series of tests demonstrated, as had been anticipated, the substantial corrosion-resistance of the austenitic nickel cast irons containing upwards of 15 per cent of nickel.

[1]Conducted at the Research Laboratory of The International Nickel Company, Inc., Bayonne, N. J.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. Of the authors, who are members of the society, J. S. Vanick is research metallurgist and P. D. Merica is assistant to the president of the International Nickel Company, New York City. Manuscript received June 24, 1930.

Realizing the possible industrial value of a really corrosion-resistant and reasonably cheap cast iron, further work was at once initiated looking to the development and refinement of this type of material. The composition of such a material is given below:

	Min.	Max.		Min.	Max.
Total Carbon	2.75%	3.10%	Manganese	1.00%	1.50%
Silicon	1.25	2.00	Nickel 12.00		15.00
Sulphur	0.04	0.120	Copper	5.00	7.00
Phosphorus	0.04	0.30	Chromium	1.50	4.00

It may be interesting to add that independent experiments along the same lines as those given in the abstract were conducted by J. Arnott of Messrs. G. and J. Weir, Ltd. of Glasgow, England, and have led him to substantially the same conclusions concerning—first, the best composition for this nickel-copper-chromium cast iron; secondly, its corrosion and heat resisting characteristics; and finally, its commercial possibilities.

Applications

One of the chief assets of this corrosion resisting iron is its low cost. Its application is useful wherever a corrosion or heat resistance superior to that of plain cast iron is necessary. In view of this, it has been made into such castings as pipe, valves, fittings, pumps, compressors, marine castings, boiler specialties, engine castings subjected to heat or corrosion, conveyors, propellors, impellers and hydraulic turbines, stove and furnace parts, oil burners, glass rolls, enameling racks, and similar types of castings going into the oil, coal, coke, caustic, sugar, glass, and other industrial or chemical operations.

It is nonmagnetic and this characteristic has led to the active consideration of its introduction in the electrical industry for such parts as transformer bushings, magneto rotors, welding machine parts, resistance grids, etc.

Its high coefficient of thermal expansion has led to its application as a sleeve in internal combustion engines; airplane, automobile and marine engines in conjunction with trunk type aluminum alloy pistons which possess a corresponding expansion under heating.

Its resistance to growth and scaling have led it into applications requiring high temperature stability, and it has substantially reduced the occurence of fire-cracks or surface checking and practically

eliminated the difficulties grouped under the failures attributed to "thermal stresses."

The ease with which this material has been handled in the foundry and shops, and the emphatic way in which it has answered the need for a cast iron of exceptional merit in its particular field, has enabled it to establish itself firmly and in daily increasing amounts for numerous applications which make use of its special properties.

Development of Nickel-Copper-Chromium Cast Iron

The early work in the development of this cast iron consisted of following the effects upon physical properties and corrosion resistance of nickel additions to plain cast iron. It was known that plain iron began to harden appreciably as the nickel content increased above 2 per cent. It was also known that at some 5 or 6 per cent nickel this hardness attained a value of 250-280 Brinell and the machining speeds gradually decreased, although the iron retained machinability and was uniformly hard and completely gray. Larger amounts of nickel continued to increase the hardness until at some 8 to 12 per cent, this factor attained maximum values ranging in the neighborhood of 320 to 360 Brinell. Still larger additions of nickel were accompanied by a pronounced softening, and in the neighborhood of 18 to 20 per cent nickel, the hardness would fall to as low as 120 Brinell, or be considerably softer than the original unalloyed cast iron. At this point, the iron was readily machinable and it possessed considerable toughness, so that it could be bent some three to five times deeper than plain iron in the transverse test. As the nickel content increased, the iron became less and less magnetic and finally ceased to be attracted by the magnet at all. This effect was naturally interpreted to indicate the complete austenitization of the material and the transformation of all of the iron from alpha to gamma. Corrosion curves illustrating the change in corrosion rate with increasing nickel over the range of zero to 20 per cent, generally showed a gradually lessened corrosion rate as the nickel content increased to something like 10 per cent, but above this and within the range of 12 to 18 per cent nickel, an abrupt decrease in corrosion rate was obtained. A further increase in nickel content, above the 18-20 per cent range, would produce a persistent though gradual decrease in the rate of corrosion. For example, in these

higher percentages, of 20 to 40 per cent nickel, a corrosion rate which in plain iron amounted to 400 mdd. would be reduced to 20 mdd. at 20 per cent of alloy while another 20 per cent would reduce the rate to perhaps 10 mdd. This indicated that in the high alloy or 20 to 40 per cent nickel or nickel plus copper range, the improvement in corrosion resistance for each increment of nickel above 20 per cent was entirely out of proportion to that obtained either in the low nickel ranges or in the range from zero to 20 per cent. For this reason, it became evident that the most pronounced decrease in corrosion could be expected in an iron containing nickel in the range of 15 to 20 per cent. Within this same range, the hardness fell off rapidly so that the iron became easily machinable again, and this factor, along with the change in corrosion rate, indicated that the best performance for the least cost could be expected in the range of 15 to 20 per cent of nickel. Fig. 1 illustrates typical hardness and corrosion curves.

In order to reduce the cost of the material still further, copper additions were substituted for part of the nickel within certain limits. These limits were guided largely by the solubility of copper in cast iron. Knowing that the copper can be made soluble in iron if it is accompanied by a suitable amount of nickel, full advantage of this fact was taken. The results indicated that the effect upon corrosion rate in many corrosives was not detrimental, and, as a matter of fact, it was found that the properties of the alloyed iron were improved in several ways. For example, the copper addition enabled a number of corrosives such as sulphuric acid, to be better resisted and its presence also increased the types of solutions which the iron might successfully resist. Some 6 per cent of copper could be accommodated in the mixture and replaced an equivalent amount of nickel, so that the resulting product was less expensive. Finally, the presence of copper lowered the melting and solidification ranges and permitted a greater fluidity in the metal, with the result that the iron retained its life practically as long as ordinary cast iron and could be cast into as thin and as intricate shapes.

Some mention might be made at this point concerning the high manganese content which is recommended in the specification. Sometime after the properties and corrosion resistance of the composition had been established, it was found that the iron showed some tendency to become slightly martensitic at temperatures around minus

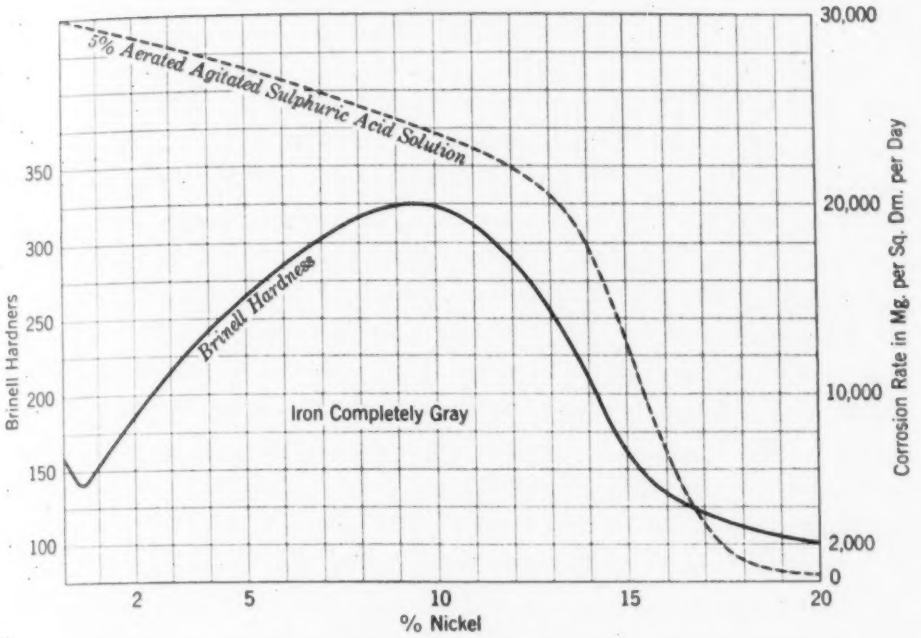


Fig. 1—Illustrating Typical Hardness and Corrosion Characteristics.

50 degrees Fahr. This unstability was registered in a tendency to become slightly magnetic and somewhat harder. Heating to room temperatures would almost eliminate the effect, and repeated cooling and warming indicated that it did not build up cumulatively, but merely affected perhaps less than 10 per cent of the total matrix. This effect was not prevalent in the higher chromium types such as those containing 3.50 per cent chromium. On the other hand, the use of this amount of chromium to prevent the low temperature transformation caused the hardness to increase excessively at the expense of machinability, as explained below. These effects were observed with normal manganese contents in the range of 0.30 to 0.60 per cent, and an investigation of the effects of higher manganese contents showed that the use of 1.0 per cent manganese in the specified composition reduced the low temperature change point to 135 degrees Fahr. below zero, and practically eliminated any risk of such a change occurring under ordinary climatic conditions. Raising the manganese to 1.50 per cent further reduced this low temperature effect to 300 degrees Fahr. below zero. For this reason, the higher manganese contents were recommended, particularly to protect equipment made of this material from a possible exposure to extremely low temperatures while the equipment was being transported through cold climates or being operated under low temperature conditions.

Mechanical Properties

The tensile properties of a 14 per cent nickel, 6 per cent copper cast iron, with the normal carbon and silicon contents, as described above, were rather low, and various expedients became necessary to make these properties comparable to those of ordinary cast iron. This can be done by lowering the silicon or carbon content or by adding chromium. The addition of chromium to the alloy appeared to be the most practical way of accomplishing this result and at the same time taking advantage of such useful properties as the chromium addition contributed. Since the iron without chromium might be as soft as 100 Brinell, the presence of something like 2 per cent chromium brought it up to 140 Brinell, so that its hardness compared quite favorably with that of plain cast iron. Larger additions of chromium continued to increase the hardness, roughly, 20 points Brinell for each 1 per cent additional chromium, so that at 6 per cent chromium, a Brinell hardness of 220-240 is obtained. This hardness is somewhat excessive for ready machinability, and a limit of 2 to 3 per cent chromium is usually recommended. The ability for the alloy to be hardened is helpful where greater resistance to wear and deformation is required in such applications as valves, pumps, impellers, feed screws, conveyors, mixers, etc. As a matter of fact, the iron can be chilled just as ordinary cast iron and a Brinell hardness of 350-400 attained. For most purposes, however, the free machining combined with good gray iron physical properties are desired and with this purpose in view a composition was standardized upon at approximately 14 per cent nickel, 6 per cent copper, 2 to 3 per cent chromium, with carbon around 3.0 per cent and silicon as in ordinary cast iron. Amsler wear tests of the brake-shoe type upon the alloyed iron of a standard composition against plain cast iron indicated that its wear resistance was nearly five times that of the plain iron under the same conditions. Naturally, wear tests require a close definition of details of procedure, but these results indicated quite definitely that the alloyed composition would wear well, presumably due to its inherent toughness as well as resistance to surface break-down under heat.

By varying the carbon and silicon composition, with other elements remaining the same, this iron will develop the following range of mechanical properties:

Arbitration bar transverse strength..... 2,500— 4,000 lbs.
Arbitration bar transverse deflection..... 0.2—0.3 inches
Tensile strength in 1¼"-section..... 20,000—35,000 pounds per square inch
Brinell Hardness..... 120—170

(Range of values is shown which depends on composition; viz., chromium, carbon and silicon contents, along with the 14 per cent nickel and 6 per cent copper.)

Attention might be called to the relatively high deflection this iron shows in a transverse test, indicating a considerable increase in toughness. This factor is further confirmed by comparative data ob-

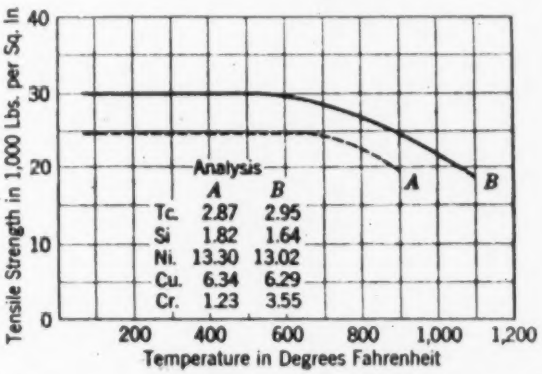


Fig. 2—Tensile Strength of Two Specimens A and B. Composition is Shown on Chart.

tained on the Izod impact machine, using a special lightweight hammer which would yield a reading on the normal scale of 80 to 110 ft.-lbs. absorbed in specimens (¼x¾ inches) of the corrosion resisting cast iron as against a reading of 9 to 15 ft.-lbs. absorbed in tests upon plain cast iron. This property has been an important factor in the adoption of the material in several installations requiring resistance to shock or impact.

Corrosion Resistance

Considerable work has been done toward determining the corrosion resistance of this alloyed iron in various corrosives both in the laboratory and in the industrial field. It is beyond the scope of this paper to include all of the corrosion data, and summaries of a few typical corrosion tests, comparing the properties of this iron with plain iron, follow:

Type of Corrosive Atmosphere	*Weight Loss in MG Per Sq. Dm. Per Day* Corrosion Resistance Cast Iron — Rusts Superficially	Plain Cast Iron — Rusts Readily
Atmosphere after 30 days	9.5	59.7
Atmosphere after 90 days	7.9	63.5
Atmosphere after 1½ years	3 to 4	30 to 40
Water Spray (Vertical)	6.6	207.5
Water Spray (Horizontal)	17.6	244.0
Aerated Tap Water Immersion	7.8	67.2
3% Aerated Sodium Chloride	50	190
12½% Fermented Molasses Solution (after 120 days)	10	360
CO_2—Sat'd* Hot Tap Water—95°C.	110	660
Ferric Sulfate	17,000	32,000
5% Aerated Sulphuric Acid	350	30,500
5% Aerated Hydrochloric Acid	507	26,665
10% Aerated Hydrochloric Acid	598	29,475
20% Aerated Hydrochloric Acid	1,111	33,270
Hot Caustic (from evaporator concentrating to 100-130 TW for 54 days	30	430

*Corresponding to bad boiler water.

Additional test data have been published in a paper by Dr. M. Marcel Ballay,[2] who had studied the material independently sometime after many of our corrosion tests had been completed.

These test results have suggested the use of the corrosion resistant cast iron in practically every service requiring a better performance than plain cast iron offers, and in some instances, the results are better than those obtained from nonferrous alloys, such as the brasses and bronzes, and in many cases, the iron can be substituted for such material, usually at a lower cost. Its resistance to attack by relatively strong acids, as well as its low corrosion rate in caustics, organic acids, weak corrosives, such as tap water, and the atmosphere, have brought it into a wide variety of industrial applications, as well as everyday uses.

Machinability

The alloyed iron if produced to the standard specification of 14 per cent nickel, 6 per cent copper, 2 per cent chromium, possesses not only the physical properties common to ordinary cast iron, but also the machinability. If the composition is hardened by chromium additions, above 3 per cent, as mentioned above (its machining speed

[2]In his Serie C, No. 4, "Recherches Sur Quelques Fontes Au Nickel et au Cuivre," in *Revue de Metallurgie*. Vol. 26, Oct. 1929, p. 538-553.

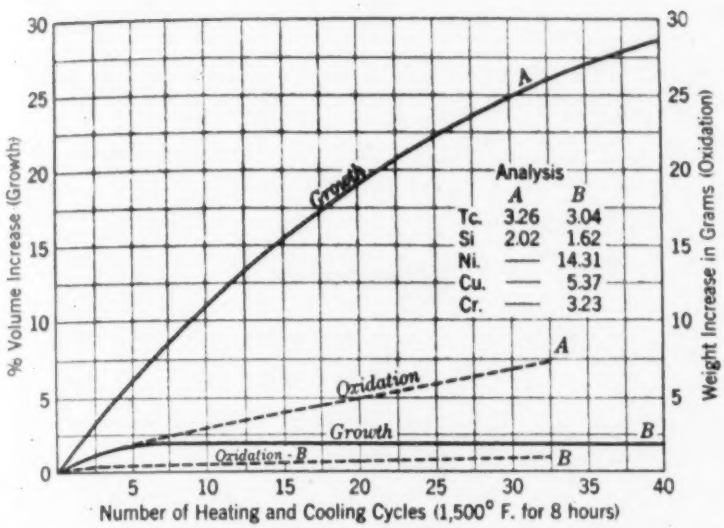


Fig. 3—Typical Curves of Oxidation and Growth of Plain Cast Iron (A) and Corrosion Resisting Cast Iron (B) at 1500 Degrees Fahr.

will greatly decrease. Hardening the iron in this way may reduce the speed to as low as one-half of that of soft gray iron. The much greater toughness of the alloyed cast iron requires that it be made softer by some 30 to 40 Brinell units to have it machine at approximately the same rate as a corresponding plain gray iron.

Thermal Expansion

The coefficient of thermal expansion for temperatures up to 600 degrees Cent. is 50 per cent greater than that of plain cast iron, and therefore similar to that of bronze. This factor has been useful in development work in connection with its use as a sleeve in automotive, aero, and marine engines, in conjunction with aluminum alloy pistons. Since aluminum possesses a high coefficient of expansion, some rather complicated piston designs have been necessary to produce a casting which would possess an expansion comparable to that of plain cast iron. By producing an aluminum alloy which expanded at about the same rate as this high expansion cast iron alloy, a combination was developed which would operate together successfully when heated. A number of engines developed along these lines in an experimental way have performed successfully and indicate the possibilities for the trend of engine manufacture in this direction.[3]

[3] "Light Metals in the Automotive Industry". Preprint A.S.T.M. Detroit Regional Meeting, Zay Jeffries, p. 13. Also S.A.E. Journal, Vol. 26, No. 6, June 1930, p. 863, "Solving Differential Expansion", by Frank Jardine.

Heat Resistance

The large amount of nickel and chromium which are present in the cast iron composition would indicate immediately that it is suitable for service at elevated temperatures. The resistance to oxidation of the ordinary composition is quite marked at temperatures in the range of 12-1500 degrees Fahr., and may reach a value showing 10 to 12 times more resistance to oxide formation than plain cast iron in an oxidizing atmosphere. As the temperature increases, this degree of improvement diminishes until at temperatures in the range of 1600-1700 degrees Fahr., the difference is considerably reduced. A curve illustrating the resistance to oxidation of a plain iron and an iron of this type is shown in Fig. 3.

A most important asset along with the resistance to oxidation is the fact that the iron is comparatively free from growth. Since the alloyed iron is austenitic in its character, and does not go through a transformation on heating and cooling with the accompanying dimensional changes, it is not so susceptible to growth as plain cast iron. Fig. 2 illustrates the difference in growth resistance between a plain cast iron and an alloyed iron of this type in an atmosphere of carbon dioxide. Further importance is attached to the fact that the resistance to growth prevails in either oxidizing or reducing gases of which carbon monoxide, carbon dioxide, steam, illuminating gas, and muffle gases, are examples.

This resistance to growth and corrosion combined with the gray iron nature of the material, may be utilized in corrosion resisting service and furthermore to obtain freedom from sticking, in metal to metal contacts.

The resistance to corrosives such as salt baths, hydrogen sulphide and other forms of oxidation or chemical attack, are indicated in the following table:

Corrosion Plain Versus Corrosion-Resistant Cast Iron in Hot Salt or Gases 1500°F.

	H_2S at 205°F.	1500°F. Liquid Salt	Oxidation Scale Formed in 240 Hours	Growth after 240 Hours Heating
Corrosion-Resistant C. I.	58.5 (x) (a)	509 (x)	9,000 (a)	2– 6% by volume (a)
Plain C. I.	319 (x)	1620 (x)	92,000	25–30% by volume (a)

(x) Wt. loss in mg. per sq. dm. per day after removing adherent corrosion products.
(a) Contained 3½% Chromium.

These results indicate a substantial resistance to chemical attack

at moderately high temperatures (not over 1500 degrees Fahr.).

Sulphur compounds and scale form less readily upon the alloyed iron and penetrate less deeply than they do in plain iron. These properties make it useful for such parts as pumps, valves, pipe, fittings, vats, pots, fans, blowers, condensers, chemical ware, and similar castings subjected to high temperature and corrosive vapours.

The strength of an iron of this type at elevated temperatures is illustrated in Fig. 2. These results have been obtained in short-time tests and indicate the usual decrease in strength as temperatures exceed 900 degrees Fahr. However, the strength loss is less than that for plain cast iron. Analyses of test bars submitted to long periods of heating, have shown that the combined carbon content remains unaffected throughout the period of heating, so that the tendency toward graphitization and strength loss which is so common in plain cast iron is not as readily developed in this alloyed type. Thus the load carrying capacity at elevated temperatures is better maintained and the rapid deterioration caused by repeated heating and cooling does not occur. This thermal stability has been responsible for several of its current uses.

Magnetic and Electrical Properties

The alloy (of normal composition) is nonmagnetic and has a high electrical resistivity (1¼ per cent of copper conductivity). Its character with respect to electrical properties is essentially that of the austenitic, high nickel, alloys. By special heat treatments or manipulation of its composition, it may be made magnetic or nonmagnetic, without changing its basic property of resistance to corrosion.

Welding

This material is unusually well suited for welding by arc or acetylene and by the use of filler rod of the same composition. The weld produced is soft and machinable with no hard ridges. It is adaptable as filler rod for the welding of plain iron as well.

Applications

This nickel-copper-chromium cast iron has been applied in general wherever a corrosion or heat resistance superior to that of plain

cast iron was required. Its corrosion resistance in general is very much better than that of plain cast iron and it will frequently equal that of the bronzes. However, its corrosion product is a rust deposit which forms very slowly and evenly over the surface. For this reason, it can not be used where freedom from rusting is essential. Its heat resistance is far superior to that of plain cast iron but it should not be expected to equal that of the high nickel-chromium types of alloys.

For some three to four years, the corrosion resistant cast iron has been used in oil refinery corrosives such as crude-oil, sludge lines, caustic lines, distillate lines, etc., largely in the form of repair castings such as valve seats, basins, pump liners, pump valves, fittings, look boxes, etc. The performance, on the whole, has been satisfactory and the quantity of castings being used in this service is increasing daily. Similar installations have been made in chemical works and in allied industries such as those dealing in sugar, paper, and food products, and in addition, such parts as filter sectors, retorts, drains, catch basins, and miscellaneous castings common to these industries, have been produced. Pumps and propellers are performing creditably in sea-water service and similar applications of this metal are in operation subjected to salt or salt-water corrosives. In the heat resisting field; glass rolls, glass annealing furnaces, and enameling racks, have been produced and in general yield six to ten times the performance of the cast iron parts which have been replaced. In some cases, stoker links, furnace parts, and similar applications, have been successfully made where the designs have limited the temperature to which the material is exposed.

Switchboard and transformer castings are in active production where advantage is taken of the electrical properties. Railway electrification and signal work has utilized a number of castings experimentally with the corrosion resistance or electrical properties in view.

Much of this iron has been in production for several years in certain places and commercial castings such as pipes, valves, fittings, pumps, and compressors, are being made by manufacturers of such products.

Acknowledgment is due a number of members of the Research Staff for their participation in this work—in particular, the help of Messrs. E. J. Bothwell, E. S. Wheeler, and T. J. Wood, with physical and corrosion tests; and D. E. Ackerman for wear test, data.

DISCUSSION

Written Discussion: By H. A. Wadman, Hartford-Empire Company, Hartford, Conn.

Some time ago I made some investigations leading to the choice of a metal, better suited than cast iron, for certain parts of a glass annealing lehr. These parts formed flues carrying hot gases at temperatures between 900 and 1350 degrees Fahr. A number of conditions, such as the production of suitable castings, machineability, price, etc., entered in determining the final selection; however, the principal quality desired was resistance to growth. Twenty-two different metals were tested including several grades of cast iron, most of the heat resistant alloys on the market, and a few welded pieces of rolled alloy, as well as structural steel and iron.

Standard test pieces of H-section ¼ inch thick and 2 inches outside were made up, 24 inches long. These were machined and then measured with a

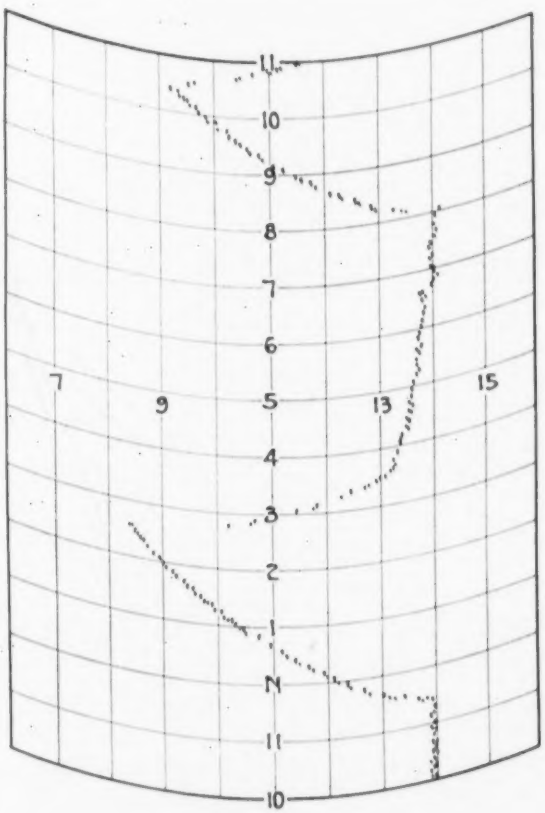


Fig. 1

vernier caliper reading to 1/1000 of an inch. They were placed in a specially designed furnace, and first given an annealing treatment by heating to 1400 degrees Fahr. for 96 hours, and then cooling to 1150 degrees Fahr. at the rate of 1 degree Fahr. per minute. The furnace was then shut down and the subsequent cooling took place at an initial rate of 2 degrees Fahr. per hour.

When cool they were removed from the furnace, any scale that had formed was scraped from the machined surface, and the pieces re-measured.

They were then returned to the furnace and heated cyclically from 900 to 1400 degrees Fahr. Each cycle required about eight hours, and these were run for periods of ten days, making about thirty cycles. After ten days the pieces were again measured, and then returned to the furnace, and the process repeated. The furnace was equipped with an automatic temperature control, and Fig. 1 (this discussion) shows the temperature as recorded at the center of the furnace by means of a recording pyrometer. Eight thermocouples were placed at various parts of the furnace. These showed that at the time the temperature was lowered, at the end of the hot portion of the cycle, the centers of the test bars varied from 1347 to 1430 degrees Fahr., while the ends varied from 1304 to 1400 degrees Fahr. The couple in contact with the test piece did not vary from a couple in close proximity by more than the variation encountered in individual couples.

Table I

No.	Fe	Cr	Ni	Si	P	S	C	Mn	Cu
1	72–70	28–30							
2	62	25	10	3					
3	52	23	23	0.5			0.8	0.5	
4	67	21	12				0.25		Welded
5	50–41	18–22	30–34				0.4	2–3	
6	16–14	18–20	65	0.6				1	
7	41	18	38	1.25–1.5			0.4–0.5	0.75–1.0	
8	44–39	17–19	37–39	1				1	
9	48	15	35	1–1.4			0.45–0.65	0.5–0.7	
10	55	14.6	30				0.26		
Nickel-Copper-Chromium Cast Iron		4.07	14.26	2.05			3.16		6.34
12	Diff.	0.5	0.3	2.0	0.28	0.1	3.3	0.65	
13	}								
14									
15	} Heat Resisting Castings								
16	}								
17	}								
18	} Stainless Steels								
19	}								
20	}								
21	} Cast Irons								
22	Structural Steel Welded								

Table I indicates the samples listed, giving the analyses as furnished by the manufacturers for the first thirteen. The iron has been set down as the difference, and the general class of material indicated in cases when the analysis is not available.

Fig. 2 shows the results of the runs, the growth or shrinkage being plotted in 1/1000 inch per 24 inches. It will be noted that the nickel-copper-chromium cast iron, Curve No. 11, shows much less change in length than No. 21, which is a very good cast iron, and that the only alloys which show changes of length of the same order are higher priced chromium-nickel alloys.

It is not contended that nickel-copper-chromium cast iron is in any way superior to, or in a higher range of temperatures, comparable to the higher

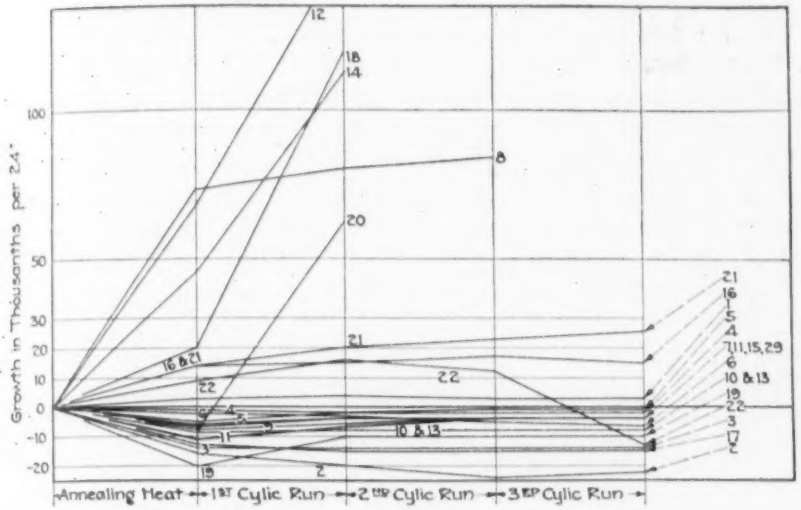


Fig. 2

chromium-nickel alloys, but its resistance to growth in the range tested is quite remarkable. Acknowledgement is hereby made to Mr. H. L. Eastman, who made the measurements and collected the data.

Written Discussion: By Harry F. Perkins, technical director, St. Louis Surfacer and Paint Co., St. Louis, Mo.

The data given in this paper does credit to the authors. The work has been intelligently carried on, there is no over statement of fact but indeed we find a spirit of conservatism.

The alloy described in this paper was known originally as Monel-Cast Iron because the nickel-copper ratio recommended is the same as in Monel metal, this makes it possible to use scrap monel metal for this alloy. Later the name Ni-Resist was coined and is the existing cognomen.

It has been my privilege to make practical corrosion tests of this alloy with very promising results. In a line carrying 30-40 degree Baume hot sulphuric acid there was only a 25 per cent loss in thickness after two years service, the line being 3-inch extra heavy pipe size. Lead is the standard metal for this purpose. Another test was made using a valve of this material placed at the bottom of an acid concentrator where the results showed the Ni-Resist to be much better than acid bronze.

Due to its toughness, fine grain and resistance to crumbling, this alloy makes excellent piston rings giving a life nearly fifteen times that of cast iron and producing less wear on the cylinder lining.

Efficient combustion requires properly constructed and regulated dampers. In large furnaces the damper in the flue often reaches high temperatures which cause them to warp out of shape, giving poor regulation, and to corrode rapidly. Asbestos, tile and chromium steels have given little better service than cast iron and cost a lot more in most cases. This new alloy Ni-Resist should pay for itself many times over in the form of dampers where it promises to outlast cast iron many times.

While Ni-Resist has performed for me in a way which coincides in general with the figures given in the paper under discussion, I have not obtained such good comparisons with water corrosion. This is probably due to the difficulty in accurately measuring the corrosion of cast iron which ordinarily does not show any deformation during corrosion and which has an awkward tendency of increasing in weight when removed from the water. We are continually reminded that cast iron does not corrode so the data here given may be a sort of "lese majesty." I have found that the comparative corrosion of Ni-Resist increases rapidly with increase in acidity of solution.

There is no difficulty in making this alloy outside of keeping the percentage of elements fairly accurate. The electric furnace is a very convenient means for handling small batches while larger amounts can be made by adding the alloying agents to the ladle pouring the iron from a cupola.

There are two classes of demand for corrosion resisting metals. One class is composed of applications where successful operation is at stake. The other class is where the problem is strictly one of economy. In the first case the cost of the metal is secondary to its performance while in the second case cost is all important, for not only must the metal justify its cost but the initial cost must remain within a limited capital expenditure and the demand in the second class is by far the greatest.

Ni-Resist is the first alloy to my knowledge that shows marked improvement over plain iron without a big jump in cost. This alloy will cost from 4½ to 6 times the price of plain iron but will give 10 to 30 times increased service, besides working easier and presenting a more pleasing appearance.

I believe industry in general will acknowledge that it owes the authors and the International Nickel Co. a debt of gratitude for this development.

Written Discussion: By B. F. Shepherd, metallurgist, Ingersoll-Rand Co., Phillipsburg, N. J.

It is necessary to closely control the composition of the iron described by Messrs. Vanick and Merica to obtain the best results. Cupola melting is none too good a tool for this. With composition much above 1.5 per cent silicon and below 2.25 per cent chromium the structure will be open-grained and the iron weak. With low silicon and high chromium considerable trouble will be found in machining.

The following physical data is given for this nickel-copper-chromium iron cast from a cupola:

									Std. A.S.T.M. Test Bar		
	T.C.	Mn	Si	S	P	Ni	Cu	Cr	TS	Trans.	Brinell
R76459	2.91	.97	1.58	.081	.125	14.59	6.35	2.45	23200	3750	118
									22400	3650	121
									22900	3800	118
R77309	3.08	1.03	1.28			12.52	5.47	2.29	21111	3500	116
									20600	3300	126
R76055	2.87	1.03	1.89	.080	.143	14.52	6.75	2.38	18200	2900	116
									18421	2700	116
									17813	3100	112
R75668	2.87	1.39	1.82	.074	.139	14.74	6.39	2.40	19438	3150	137
									19839	3000	137

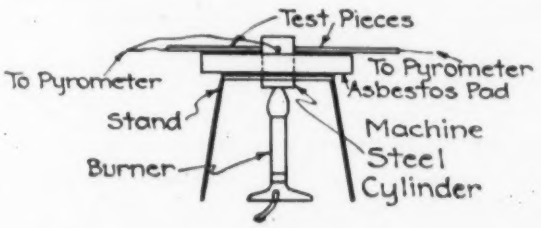
Salt spray tests were made on standard navy specimens comparing Ni-Resist with gray cast iron. The time of test was 100 hrs.

Ni-Resist — .0671% loss in weight average 2 samples
1.10% Si Cast Iron—1.04 % loss in weight average 3 samples
2.00% Si Cast Iron— .791 % loss in weight average 3 samples

The results of tests to determine the heat conductivity of Ni-Resist cast iron as compared with the following cast irons is shown in the accompanying curve.

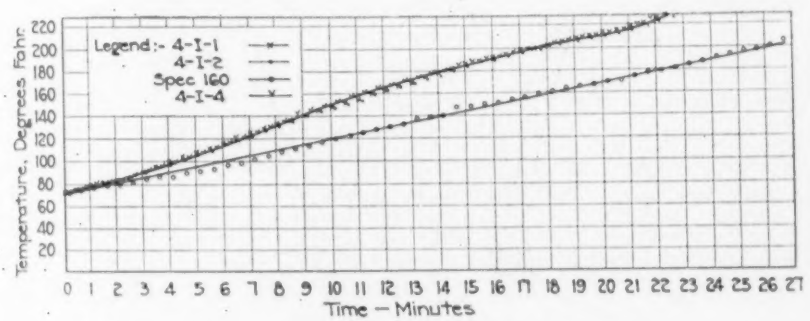
4-I-1 1.10 per cent silicon 35 per cent steel in mixture.
4-I-2 1.70 per cent silicon No steel in mixture.
4-I-4 1.70 per cent silicon 35 per cent steel in mixture.

The samples used were ⅜-inch round bars, 4 inches long, screwed 90 degrees apart into the middle of a steel cylinder 1¼ inches in diameter, 2 inches



long. A 1/16-inch hole was drilled into the outer ends of the sample for thermocouple leads. The effective length of the samples was four inches.

The cylinder was set into a hole in a heavy asbestos pad and a Bunsen burner flame applied (see sketch). Temperature readings were made for each



bar every minute. The results show the Ni-Resist conducted heat at a slower rate than our standard cast irons. The heat conductivity of the three plain cast irons was practically the same.

R. S. ARCHER:[1] The authors have covered in a brief but complete way the story of the application of this iron in connection with aluminum pistons. The most common aluminum piston alloys (10 per cent copper) have a coefficient of expansion of about 0.000022 per degree Cent. The new alloy which is referred to on the ninth page of the paper has an expansion

[1]Research laboratories, Aluminum Company of America, Cleveland.

coefficient of about 0.000019 per degree Cent. This is almost the same as that of the special nickel-chromium cast iron which, I believe, is about 0.000017 or 0.000018. As the authors have stated, the combination of nickel-chromium iron and the low expansion aluminum alloy has been used successfully in an experimental way. There is not much to add to this, except to confirm it. The piston engineers of the Aluminum Company have been quite active in the engineering of this combination, and where this has been used the results have been strikingly good. It is a matter of incidental interest that the special low-expansion aluminum alloy also contains nickel.

GLEN COLEY:[2] I would like to ask the author if he has done work on this steel relative to the so-called creep tests at temperatures of 1100 to 1200 degrees Fahr.

P. R. LETZ:[3] The authors are to be congratulated on doing this work on cast iron. As we all know, we needed some work of this kind.

About one year ago, the company of which I am a member was requested to design a compressor unit for natural gas rich with hydrogen sulphide. There are, of course, a good many materials available that give very satisfactory corrosion resisting properties, but their cost is high, particularly if it is necessary to make new patterns. Machining of these alloys is difficult, and therefore also expensive. It occurred to us that it is possible to use high chromium-nickel-copper cast iron for the purpose. In order to determine the adaptability of this alloy for the purpose, we carried on a number of tests in our laboratory, in which we tried the corrosion properties of the special iron, comparing it with the various alloys available on the market, and with ordinary iron. We found that corrosion properties of this alloy are very satisfactory in an atmosphere of hydrogen sulphide gas, as well as an atmosphere of hydrogen sulphide gas with steam vapors and oxygen. As a result of this test, several compressors of fairly large size were built by us, about 18 to 20 inch bore. They are now in service. However, they have been in service such a short time that we have not had an opportunity to obtain data which would be worth while to present here. Nevertheless, reports indicate that they are very satisfactory, and the possibility of using this iron for the compressors is very good.

H. A. SCHWARTZ:[4] If the authors have anything to add along the general line of wear resistance, both of the type where the wear is caused by the cutting of abrasive material and of the type where stresses are heavy enough for austenite to work harden, I will be very grateful.

Authors' Closure

There is nothing to be replied to in the discussions contributed by Messrs. Wadman, Letz, and Archer. We keenly appreciate the facts they bring forth. Also those from Mr. Perkins, who during his connection with one of the large oil refiners initiated corrosion tests of the material in the laboratory, field and

[2]Metallurgist, Detroit Edison Co., Detroit.

[3]Cooper-Bessemer Corporation, Mt. Vernon, Ohio.

[4]Manager of research, National Malleable and Steel Castings Co., Cleveland.

refinery, and really started its commercial production for the oil trade. His reference to ladle alloying obviously implies the addition of alloying agents in liquid form because the addition is too large to make unless thoroughly heated. Again, the reference to cost differential between this iron and plain iron apparently refers to cast iron pipe where the metal cost is the most expensive element in the cost of the product. At present, the raw metal cost of the alloy addition amounts to approximately 6 to 7 cents per pound and quotations brought to our attention from foundries producing quantity castings, indicate finished castings quoted for 10 to 15 cents per pound over plain iron casting costs.

Replying to Mr. Coley, we have not made creep tests. However, this iron does not graphitize under heat, and should retain its load-carrying capacity over a longer period than plain cast iron. Heating cast iron to 1500 degrees Fahr. for 60 days and slowly cooling causes a strength loss in plain iron of 20 to 50 per cent when tested at room temperatures, whereas the nickel-copper-chromium iron retains its original strength after such treatment.

On the seventh page are some quick tensile tests which illustrate the type of curve you will get, and can probably be extrapolated in the light of knowledge we now have on creep to give you some idea about where these irons will go.

With reference to applications in stoker parts, when allowance is made for the expansion, or extra clearance is made in the fire box or places of that sort for this kind of iron, it performs very well. It has been used in stoker tuyeres and also in waffle irons, in lining fire boxes. If the temperature does not get too high, the performance is fairly good. By too high a temperature I mean temperatures over 1650 degrees Fahr.

Replying to Mr. Schwartz—resistance to abrasion has usually been developed by hardening with the aid of low carbons, low silicons, high chromium, chilling, or all together. Its resistance, then, compares well with plain chilled cast iron but its application has been useful where equipment which is subject to abrasion and corrosion, is corroded during its idleness or attacked by corrosives along surfaces which cannot be protected.

Work hardening does occur in the usual nickel-copper-chromium composition, and sand blasting rather than tumbling is recommended for cleaning castings. The skin of poppet valve seats in engines and a honed cylinder is liable to develop a thin, work hardened case. The actual performance in engines has been better, from the wear standpoint, than that of cast iron; one manufacturer reporting that the wear after 18,000 miles, was no greater than that after 8,000 in their usual alloyed cylinder iron. Bearings or bushings are being made of the material for sliding or rotating contacts and in this service a soft composition, brinelling approximately 120 to 140, is used, in which the chromium content is 1 per cent or less.

The Amsler tests mentioned in this paper refer to a comparison of various cast irons used as "shoes" against "wheels" or disks of the nickel-chromium-cast iron brake drum composition. Pressure was 125 pounds per square inch of brake-shoe surface on 40 millimeter diameter disks rotating at a speed of 210 revolutions per minute.

Mr. Shepherd's discussion arising from results obtained in experimental production of the alloyed cast iron is very useful. It emphasizes one point; namely, that the differences in cupola practice (such as the use of high or low silicon pig, percentage of steel, quantity and shape of "Ni-Resist" scrap and foundry scrap, etc.) and numerous contingent variables which govern the melting procedure for each shop produce their corresponding effects. The development of best physical properties in irons apparently identical in chemical composition is a problem which each foundry or cupola producer needs to solve individually.

BEARING METALS AND BEARINGS

By W. M. Corse

Abstract

A brief historical survey is given, tracing the development of bearings and bearing metals. This is followed by a discussion of the requirements of a good bearing metal and a description of the properties of white metals, bronzes, and graphite bearing metals.

The utility of the tests most commonly applied to bearing metals is discussed and a summary given of some recent important work on bearing bronzes at the Bureau of Standards.

The author takes up the various factors to be considered in the design of bearings and the causes of failure.

A brief non-mathematical resumé is given of the theory of lubrication, both for thick and thin lubricant films, referring to recent research work in this field. The effects of various physical and chemical properties of the lubricant are discussed and further fundamental research recommended.

A BEARING as we understand the term is a mechanism devised to reduce to a minimum the friction between rotating or sliding surfaces in contact with each other. The bow-string drill still used by the Indian jewelers of the Southwest was invented before the dawn of history and probably used the first thrust bearing, while the first radial bearings were no doubt those of the ox-cart, with its solid wooden wheels.

Wood bearings were satisfactory so long as there was no need to move loads rapidly. But when horse-drawn chariots came into use it was necessary to use metal-lined bearings. Horse-drawn chariots were used in the Trojan War, 1100 B. C., and whole squadrons are depicted on the memorials of Rameses II, 1300 B. C. Czochralski and Welter state that iron bearings were in use in ancient China and India.

A paper presented before the Detroit chapter of the society, November, 1929. The author, a member of the society, is consulting metallurgical engineer, Washington, D. C. Manuscript received March 8, 1930.

However, the Middle Ages found engineering in a Dark Ages of its own and very crudely constructed wooden bearings were being used. Early in the 15th century we find a renewed development in engineering fields, and reproductions of old drawings of about 1588 show the shaft of a water wheel resting on a wooden block held in place by an iron strap bent to fit the shaft, while the forerunner of the present overhead lineshaft bearing is illustrated by a shaft rotating in a hole through a wooden block reinforced by an iron strap.

With the development of the turning lathe, in the 18th century, bearing construction received an impetus, but very diverse opinions were held as to what constituted a suitable bearing material. Wooden bearings were lined sometimes with iron, sometimes with brass or bronze, and others with leather or fabric.

In order to meet the engineering demands of today a good bearing metal must be able to carry the weight of the shaft and stand up under steady or suddenly applied loads, or even blows in some types of service. A certain amount of plasticity is desired to compensate for minute irregularities in the shaft or bearing surface, and to permit yielding, thereby avoiding dangerous stresses in bearing and shaft and preventing overheating and rapid wear of the parts. Less plasticity is required in a bearing metal if the surfaces of bearings and journals are machined accurately, aligned, and kept in alignment. It is desirable, of course, that a bearing metal have a low coefficient of friction. Under most operating conditions the friction depends on the unit pressure of the bearing, the speed, the temperature, and the properties of the lubricant.

A good bearing metal must have a low rate of wear to reduce the necessity for frequent replacements, and adjustment of the bearings. It must wear more rapidly, however, than the journal or shaft running on it, because replacement of the journal or shaft is more difficult and costly than replacement of the bearing.

In many cases a bearing metal of good running properties is not strong enough to support the load by itself, but is used as a comparatively thin lining in a shell of some strong and cheap material such as cast iron, steel or bronze. On the other hand, a strong and hard material is sometimes desired for a bearing metal, particularly where accuracy is required. In such cases both shaft and bearing metal must be carefully machined, and finished by scraping, grinding or lapping.

History of Development of Bearing Metals

Special alloys for bearing purposes were first introduced between 1792 and 1817, and the rapid progress of mechanical engineering since that time has been greatly aided by the development of better bearing metals, improved bearing design, better lubricants and methods of lubrication.

Alloys produced in the 18th century and the early part of the 19th century were suitable for bearing purposes from the point of view of running quality, but they were not strong enough to support heavily loaded shafts without squeezing out. A wider use of these alloys became possible, however, when Isaac Babbitt used successfully a soft metal lining in a harder and stronger shell. Babbitt used a comparatively soft tin-base white metal but did not restrict the claims of his patent to any particular composition.

A soft metal lining in a harder shell permits load carrying capacity to be increased with no sacrifice of the plasticity which permits the bearing surface to adjust itself to the contour of the shaft, or to slight lack of alignment. This plasticity was lacking in the tin bronzes used in the early days of railroading, and hot boxes were common because of faulty adjustment.

Hopkins lined bronze bearings with thin sheet lead in 1870, but he found this too plastic. Antimonial lead, which is harder, proved to be more satisfactory.

The need for a more plastic metal than bronze or cast iron suggested the idea of lining bearings with soft metal. But little progress was made for some time toward improving the plasticity of bronze or other hard alloys, in order that satisfactory bearings could be made entirely of the same kind of metal. Developments here and in England have been toward the production of a bronze containing considerable amounts of lead to increase its plasticity.

Much investigation was necessary to discover satisfactory methods for making and founding of the lead bronzes. About 1870 Alexander Dick invented the alloy copper 80, lead 10, tin 10, which was the standard bearing metal on British Railroads for years. In 1892 C. B. Dudley in the United States produced Ex. B metal, copper 77, lead 15, tin 8, and his tests of actual bearings in service showed that the rate of wear and tendency to overheat diminished with increase of lead content, and increased as the tin content increased. Ex. B metal became the standard bearing metal on the

Pennsylvania Railroad, replacing their Standard S, which was the same as Dick's Bronze.

In 1900 a patent was granted to G. H. Clamer and J. G. Hendrickson for a series of lead bronzes containing over 20 per cent lead and less than 7 per cent tin. Their invention was based on a scientific knowledge of the properties of copper-tin alloys.

Clamer and Hendrickson's invention consisted in limiting the ratio of tin to copper to 9 to 91, so that freezing took place quickly and trapped the lead before it had a chance to sink through a partially solid metal which could not be stirred. Other ways were found to make high-lead bronzes, notably the method of A. Allan, Jr.

Allan's method involves the use of sulphur, which diminishes the temperature range in which molten lead and copper are immiscible, so that segregation difficulties are less aggravated by segregation in the liquid state. In all probability the knowledge of the effect of sulphur was arrived at accidentally, and not through the results of scientific investigation as in the case of Clamer's alloys.

The lead bronzes now find wide applications on the railroads and in stationary machinery where they stand up under severe conditions. Specifications for six standard lead bronzes were drawn up by the American Society for Testing Materials in 1921 and these alloys are now being used in the automotive industry.

Types of Bearing Metals

Three types of bearing metals have been selected for discussion: White metals, bronzes, and graphite bearing metals.

White Metals

The general requirements of a white metal for bearing purposes are that it should give good adhesion to the shell, have high compressive strength at working temperatures, good wearing qualities, low coefficient of friction, and finally sufficient plasticity to compensate for any inequalities in alignment.

The ordinary white bearing metals are essentially alloys of lead, tin, anitmony, and copper. Lead or tin is usually the major constituent. The high tin alloys are superior mechanically but are the most expensive. Tin and lead harden each other somewhat but straight lead-tin alloys are not suitable for bearing metals. The

addition of antimony causes an increase in the hardness of both of these metals and their alloys.

Not all of the alloys of this system are suitable for use as bearing metals. Those containing over 25 per cent antimony are too hard and brittle, and those containing less than 5 per cent antimony are too soft and yielding. A measure of the plasticity, that is the ability of the bearing metal to adjust itself to the contour or alignment of the journal, is shown by the amount which a specimen of the alloy may be compressed before cracking.

Many of these alloys contain copper, in addition to lead, tin and antimony. The copper is added primarily to prevent segregation, though it also improves the mechanical properties somewhat by stiffening the groundmass. If no copper is present the first constituent to crystallize out of the molten alloy on cooling is the compound SbSn, which forms cubical crystals. These crystals, being lighter than the melt, rise and segregate, unless special precautions are taken. If more than 1 or 2 per cent of copper is present, the first constituent to crystallize is a copper-rich one, containing Cu and Sn in the tin-base alloys, and the compound Cu_2Sb in the lead-base ones. Both of these constituents crystallize as needles and form an interlocking network which entraps the subsequently formed cubes of the SnSb compound and prevents them from rising.

Alloys containing less than 10 per cent tin should contain very little copper. The specifications of the American Society for Testing Materials limit the amount of copper in such alloys to 0.5 per cent, while in the German Industrial Standards (D. I. N.) alloys the copper content is set between 1 and 2 per cent for those containing 10 per cent tin or less. Experiments at the Chicago Bearing Metal Co. showed that "the addition of only a few tenths of a per cent of copper to such an alloy as Pb, 82-94; Sb, 9-11; Sn, 6-8, will cause it to froth on pouring at any temperature below 482 degrees Cent. (895 degrees Fahr.). Most of the copper may be removed from such an alloy by sweating." These alloys, according to Bauer have very little tendency to segregate anyway, therefore the copper is not necessary to prevent segregation.

There is some difference of opinion as to proper pouring temperature. It will vary somewhat with the wall thickness of the casting to be made, and the temperature of the mold. The general rule for pouring these alloys is to pour at the lowest temperature at which the metal will completely fill the mold. E. R. Darby has

shown that the lead-base alloys maintain their fluidity almost down to their melting points, while the tin-base ones lose fluidity some 100 to 150 degrees Cent. (210 to 300 degrees Fahr.) above their melting points. Darby's fluidity measurements showed that the pouring temperatures given by the American Society for Testing Materials, for the standard white metal bearing alloys, while lying near the points of maximum fluidity in some cases, were considerably too low in other cases. This was especially true of alloy No 5, composition Sn, 65; Pb, 18; Sb, 15; Cu, 2; for which Darby found the lowest permissible pouring temperature to be 500 degrees Cent. (930 degrees Fahr.), instead of the specified temperature of 365 degrees Cent. (690 degrees Fahr.). Pouring temperatures of the American Society for Testing Materials lead-base white metals lie between 325 and 375 degrees Cent. (620 and 710 degrees Fahr.), while pouring temperatures of the tin-base ones lie between 440 and 500 degrees Cent. (825 and 930 degrees Fahr.). Czochralski and Welter give 400 degrees Cent. (750 degrees Fahr.) as the pouring temperature both of Sn, 80; Sb, 15; Cu, 5; and Pb, 80; Sb, 15; Sn, 5.

The advantages and disadvantages of the lead-base and tin-base white metals may be stated briefly as follows: The tin-base alloys are superior mechanically, those containing about 80 per cent tin possessing maximum strength and hardness (compressive strength about 20,000 pounds per square inch; Brinell hardness about 28). The lead-base alloys are much softer and less strong (compressive strength 13,000 to 15,000 pounds per square inch, Brinell hardness 15 to 20). Since the high price of tin increases the cost of the tin-base alloys, advantage is not taken of their better properties as it is more economical to use a lead-base alloy if the design permits. However, where economy of space and weight are essential, as in aircraft engines, the tin-base alloys will continue to be used as the same load capacity can be obtained with smaller bearings. Another disadvantage of the tin-base alloys is their low fluidity near the melting point which makes it necessary to pour them at temperatures of about 100 to 150 degrees Cent. (180 to 270 degrees Fahr.) higher, at least for thin linings, in spite of the fact that they melt at about the same temperatures as the lead-base alloys.

White metals made wholly or partly from scrap metal often contain impurities, the most harmful of which are zinc and aluminum; the amount of these impurities is limited to zero in the American Society for Testing Materials specifications for white metals.

Other impurities are iron, arsenic, and phosphorus.

In view of the high price of tin, and the difficulty of obtaining it at times, a condition that faced the United States during the War, much work has been done to discover other alloying ingredients to improve the properties of the lead-base alloys.

These new alloys consist of lead hardened by the addition of small amounts of alkali and alkaline earth metals. In this connection it was discovered that a small amount of sodium gave the same hardness as a much larger amount of antimony. "Noheet" containing 0.5 to 4 per cent sodium was patented in 1905. It had satisfactory properties as a bearing metal, but it was subject to atmospheric corrosion.

In 1915 Frary and Temple (U. S. A.) invented a series of alloys of lead hardened with small amounts of the alkaline earth metals. Some of these, such as "Ulco Metal" or "Frary Metal", were found suitable for bearing linings. Similar alloys known as Mathesius Metal and Lurgi Lagermetall were developed in Germany about the same time by Walter and Hans Mathesius; these have been covered by patents in the United States. Lurgi, which was the standard lining metal on the German railroads for some time, has been succeeded by Bahnmetall whose resistance to atmospheric corrosion is greater owing to the addition of 0.05 per cent lithium.

Two methods have been followed in the attempt to improve the properties of the lead-base alloys, namely:

1. Improving the strength of the soft lead-rich matrix by adding a substance which forms a solid solution with it, and

2. Increasing the strength of the other constituents embedded in the lead-rich matrix, or even finding new and better hard constituents.

The first method is exemplified by Thermit bearing metal in which the matrix of a bearing metal containing 72 to 78 per cent lead is strengthened by the addition of 0.7 to 1.5 per cent nickel, 0.7 to 1.5 per cent cadmium. The compressive strength of this alloy is 25,600 pounds per square inch, and the Brinell hardness 29, so that it is somewhat superior mechanically even to the high-tin alloys.

The second method is exemplified by the lead alkali and lead alkaline earth metal alloys. Here new hard constituents are formed which are intermetallic compounds of lead with the added metals. The best example of this type is probably Bahnmetall which, as mentioned above, is a standard lining metal today on the German

National Railroads. It has a compressive strength of 25,000 to 30,000 pounds per square inch, and a Brinell hardness of 34. The hardness of cylindrical specimens is accompanied by a good degree of plasticity. The metal may be compressed 28 per cent without cracking. Bahnmetall contains 0.73 per cent calcium, 0.04 per cent lithium, and 0.58 per cent sodium, balance lead.

Satisfactory bearing metals can be produced from lead alkaline earth metal alloys without using tin, and the small amounts of alloying ingredients required add little to the cost. However, these alloys have the disadvantage of requiring special methods of manufacture and suffer loss of their alloying constituents on remelting. Their pouring temperature is high (500 to 600 degrees Cent.) and most of them are more or less susceptible to atmospheric corrosion, although this fault is said to have been eliminated in Bahnmetall by adding a small amount of lithium.

While linings can be cast of these metals in properly equipped bearing metal foundries, it is a special problem to make them up from their components. This is usually accomplished by electrolysis of a mixture of fused alkali and alkaline earth metals using a molten lead cathode.

Bronzes

Under some conditions soft metal linings are not always suitable and bearings are made entirely from bronze, cast iron, wood, or other materials. Even where bronze is used on top of cast iron, as in a die-cast liner or bushing, it is not spoken of as a lining metal. Most bronzes used today for bearing metals contain considerable amounts of lead to give them that degree of plasticity which will compensate for slight want of fit or alignment of the bearing. Lead-containing bronzes are also used for supporting white metal linings when a somewhat plastic backing is desired. For instance, red brass containing lead is used as the standard backing metal for Bahnmetall or other white metal on the German National Railroads. Where accurate fit and alignment can be maintained, it is permissible to use lead-free bronzes, usually phosphor bronzes. These are strong, very hard, and have a low coefficient of friction. They will wear rapidly, however, and will overheat and score the shaft if poorly fitted or if the supply of lubricant fails.

The ordinary bronzes consist of a copper-rich solid solution

with tin, in which is suspended the so-called "delta" constituent—a hard compound of 21 per cent tin. If more phosphorus is present than needed to deoxidize the metal, the compound Cu_3P may be present. Both this compound and the delta constituent are harder than soft steel. Therefore care must be taken not to use too soft steel journals against bronze. Also if the metal has been burnt in founding, crystals of tin oxide—one of the hardest substances met with in alloys—will be present, and these always score the journal.

The lead bronzes contain lead held mechanically in a matrix of tin bronze, very little of the lead being in solid solution. Some recent work at the Bureau of Standards indicates that the following results are applicable to bronzes containing tin and lead:

"1. Alloys containing less than 4 per cent tin and 4 per cent lead have very high wear rates. Increase in either tin or lead over this amount decreases the wear rate. The rate of this decrease is slow with increases in tin or lead over 7 per cent.

"2. Alloys with high tin and low lead have high friction. As lead increased or tin decreased or both, the friction becomes progressively less.

"3. Alloys with less than 4 per cent lead develop rough wearing surfaces in unlubricated wear. Alloys high in tin develop rougher surfaces than those low in tin. Increase in lead or decrease in tin results in progressively smoother surfaces.

"4. Resistance to pounding is governed by ratio of tin to copper. The higher the ratio of tin to copper the better the resistance to pounding. Increase in resistance to pounding may be obtained by increasing lead as when copper is reduced the ratio of tin to copper increases.

"5. Increase in either tin or lead decreases the toughness. The effect of lead is a gradual decrease while the effect of tin is gradual up to about 8 per cent. When the tin is increased over 8 per cent the alloys become very brittle.

"6. Hardness—The hardness of the alloys is largely effected by tin content and very slightly by lead content. Hardness is not a measure of resistance to pounding."

The lead bronzes, like all mechanical mixtures of two metals having different specific gravities and different melting points, are liable to segregation. This tendency is further aggravated by the fact that copper and lead are immiscible in the liquid state unless the temperature is considerably above the melting point. The addi-

tion of sulphur diminishes the range of immiscibility and thus aids in the prevention of liquation.

Graphite Bearing Metals

The graphite bearing metals are, in fact, bronzes or white metals mixed with graphite, the latter being a material possessing negligible strength, and usually classed as a lubricant. The graphite in an alloy is held mechanically in a state of fine dispersion. Its function is to absorb the lubricant in relatively large quantities. Therefore, graphite bronze bearings frequently do not need lubrication for a long time. This is a decided advantage in enclosed machinery, such as motors.

The graphite may be incorporated in the molten alloy under pressure, as in the case of Graphalloy, or by sintering the partially reduced oxides of the metallic constituents with graphite under high pressure, as in Genelite or Durex.

Most of the graphite alloys are porous, serving to hold the lubricant as noted above, or, in some cases, so porous that the lubricant may be supplied through the bearing metal itself by capillarity. Porosity, however, is accompanied by mechanical weakness, so that the alloys serve only as small bushings or linings.

Types of Bearings

There are two basic types of bearings, namely sliding contact bearings and roller contact bearings. Each type is divided according to the manner in which it takes the load, into (a) radial bearings, where the bearing opposes forces applied perpendicular to the axis of the cylindrical shaft; (b) thrust bearings which take loads parallel to the axis of the shaft, i. e., end thrusts; and (c) angular contact bearings which take both kinds of loads.

Sliding contact bearings may be either plain bearings, in which the bearing surface is continuous except for oil grooves, or pivoted-block bearings such as the Kingsbury and Michell, which are used almost exclusively today for taking large end-thrusts. In the case of sliding contact bearings, bending stresses in the shaft must be taken care of by plasticity of the bearing metal, or by making the whole bearing self aligning.

Roller contact bearings may be either ball or roller bearings, the latter using either cylindrical or tapered rollers. Ball bearings are now made which allow quite a large departure from alignment.

Factors Deciding Choice of Type

The first cost of rolling contact bearings is for the most part greater than that of most sliding contact bearings to which they are equivalent, as they are usually made of expensive materials such as high grade alloy steels, and the operations of forming, machining and heat treatment are expensive.

The pivoted block types of sliding contact bearings are naturally more expensive than plain ones of the same size, but they can carry a load many times as great, and carry it more reliably.

Sliding contact bearings are made of a great variety of materials with corresponding variations in cost. In white metal bearings, the cost is determined largely by the amount of tin in the composition.

The prospective user will do well to consider carefully the use to which bearings will be subjected before making a selection.

Bearing Design

As the object of a bearing is to eliminate as far as possible friction between two moving surfaces in contact with each other, bearing design is of major importance to the engineer.

When designing bearings one must consider the materials in the bearing itself, the material of the shaft, in the case of journal bearings, and, finally, the lubricant which tends to reduce the friction.

Reduction to a minimum of the following economic losses depends on the proper design of bearings, and on the choice of materials and lubricants:

1. *Loss of Power.* The greater the bearing friction, the greater the amount of energy converted into heat, which raises the temperature of the machine, or part, disastrously if not carried away faster than it is developed.

2. *Wear.* Operation of machinery is accomplished by wear of the bearing surfaces in two ways, i. e.,

(a) uniform wear which can be compensated for by adjusting the bearing from time to time, or

(b) scoring, which necessitates machining of worn parts, and in many instances replacement of the damaged part.

Wear involves not only the loss of the metal itself, but the cost of the labor employed in constructing the bearing, because it becomes worthless and must be discarded or repaired before a large portion of it is worn away. Excessive wear results in a general

depreciation in the value of the machine, because loose bearings, if not taken up, lead to excessive vibration or pounding with consequent loosening, fracture of themselves, or of other parts of the machine. Rapid wear results in high maintenance costs.

3. *Consumption of Lubricant.* Even when circulation is provided for the lubricant, there is waste either by leakage, evaporation, or chemical and physical changes which take place in the lubricant, thus gradually unfitting it for further use.

The first step in designing a bearing is to determine what overall dimensions are required to support the load under the conditions of operation of the machine. The projected area of the bearing must be great enough to:

(a) Prevent the bearing metal from flowing out under the load, and (b) Maintain fluid lubrication under operating conditions.

The first requisite depends on the mechanical strength of the bearing metal, its frictional and wearing qualities, as well as on the accuracy of alignment and fit which it is possible to obtain in constructing the machine, while the maintenance of fluid lubrication depends on the distribution of the lubricant in the bearing and the correct design of the oil grooves. Such design should be based on a knowledge of the distribution of pressure within the bearing.

Incorrectly designed grooves are much worse than none at all, and may prevent the establishment of fluid lubrication under any conditions. The points of entrance of the oil should be so located that oil is drawn into the bearing by the rotating shaft, that is, from regions of low to those of high pressure. Sharp edged grooves are to be avoided as they tend to break the oil film. Grooves should not extend to the ends of the bearing, as this would not allow pressure to be built up in the oil film.

No detailed directions for the design of oil grooves can be given here, and the reader should study the practice of reliable machine builders. The practice of the Westinghouse Machine Company is given by figures and a table in Marks Mechanical Engineers Handbook.

Bearing Troubles and Failures

Bearing troubles and failures may be traced to one or more of the following causes: Faulty design of bearing; materials unsuited for the service, improper lubrication, and incorrect methods of manufacture.

Faulty Design. The design of bearings is interconnected with the design of the machine as a whole. Thus if flexure of a shaft occurs, either the shaft must be made stiffer, or the bearing made self aligning. If flexural stresses occur in a shaft that is not stiff enough, and the bearings are not self aligning, fracture of the bearing lining or of the shaft itself may take place, if the bearing is too heavy and rigid. On the other hand, if lighter bearings are used, excessive flexure may occur, resulting in destruction of the bearing or damage to other parts of the machine. For instance, flexure of a shaft carrying gears may lead to uneven wear and breakage of the teeth. Rapidly alternating stresses may cause loosening of linings in bearings which are not rigid enough, even when the maximum value of the stress is below the yield point of the material. The choice of the dimensions of the shaft or journal is just as important as that of the bearing, and the two must be considered together, because if the shaft is too small for the load no bearing will give good service. The effect of fatigue is especially to be guarded against in designing bearings, on which the load varies in amount or direction.

Overheating of a bearing may be caused by too small clearance resulting in friction or imperfect distribution of the lubricant. On the other hand, there may be too great a clearance with consequent failure to maintain fluid lubrication. While satisfactory dimensions and clearances for bearings can be found in standard engineering handbooks, so that bearings as built generally give satisfactory service unless of unusual design, the clearance may be allowed to wear too great in service, or it may be too small when the bearing is refitted, and the results are equally disastrous.

Bearing design is sometimes faulty as to point of entrance of the oil and as to grooving. The pressure in an oil film may be many times that of any oil supply system, therefore bearings in which the oil entrance is at a region of high pressure will fail to give good service. In such bearings, a fluid film cannot be built up, and the bearing then operates under conditions of partial lubrication. The oil grooves may be so constructed in bearings that regions of high and low pressure are connected and the otherwise possible pressure difference cannot be built up. Grooves with sharp leading edges are undesirable in that they tend to break the film, especially under conditions of thin film lubrication. Oil rings sometimes fail to deliver the necessary amount of lubricant and overheating follows.

Unsuitable Materials. The bearings as a whole must be strong enough to support the load and any bending stresses in the shaft, as well as to prevent the bearing metal from cracking or flowing out under the maximum pressure in the bearing, due to the load on the shaft.

Bearing metals may give poor service because of unsatisfactory wearing qualities. These qualities depend on the properties of the several constituents, and may be greatly affected by small amounts of impurities. If standard alloys are used and the impurities kept below the limits given in the specifications, little trouble will be experienced from this source.

Lubrication. Many bearing troubles are caused by unsuitability or deterioration of the lubricant. The lubricant chosen should be one which will give good service under conditions of partial as well as perfect fluid lubrication. New oils should be watched carefully in service, and changed when signs of faulty lubrication appear. Oxidation is one of the chief causes of deterioration of oils, though there is some evidence indicating that some oxidation is necessary. In automobile engines, oil may become diluted with gasoline, or contaminated with carbon. In turbines, emulsification of the oil with water is the greatest danger.

Incorrect Methods of Manufacture. Many bearing troubles are due to the metal being improperly cast or treated improperly after it has been cast. White metals cast at too high temperatures, and too slowly cooled, have a coarse grained structure and are unsuitable for bearing metals. On the other hand, if they are too rapidly cooled and quenched as soon as solid, they will be too fine grained for bearing metals. Lead bronzes, however, must be cast at rather high temperatures to prevent segregation.

When bearings are lined with white metal it is desirable that the backs should be heated to avoid shrinkage strains due to unequal contraction of the two metals after the lining becomes solid, because such internal stresses may cause the lining to crack in service. Once the lining is loosened from the back, oil creeps through the apertures and being a poor conductor of heat the temperature of the bearing may rise to a dangerous point. If the lining cracks, the pressure of the oil may be instrumental in loosening it, if it connects with spaces between the lining and the back.

Oxidation of bearing metals when in the molten state is always to be avoided, as some of the products of oxidation are particularly

deleterious. The worst of these is tin oxide, which may occur as diamond shaped crystals in both bronzes and white metals. It is extremely hard and will score a hardened steel journal.

Testing Bearing Metals and Bearings

Static Tests. The static mechanical tests that are of importance in testing bearing metals are (1) the compression test, (2) the hardness test, and (3) the transverse bending test. The tensile test is of slight importance, as bearing metals are rarely under tension in service.

In compression tests the size and form of the specimen are of importance. As many nonferrous metals have no well defined elastic limit and yield point, nor do they always fail by shearing off or cracking, it is necessary to give arbitrary definitions of elastic limit, yield point and ultimate compressive strength. It will be best to use the definitions given by the American Society for Testing Materials in the results of their tests on white metals and to use specimens of the same dimensions as they used. The per cent compression under some definite load, or at the beginning of cracking are also sometimes of value.

Hardness Tests. Practically the only hardness test of value for bearing metals is the Brinell or some modification thereof. Some bearing metals are a too coarse-grained mixture of hard and soft crystals for the scleroscope to give good results. Some work has been done on instruments designed to measure the hardness of the individual constituents, such as the Bierbaum microcharacter. Such instruments require a carefully shaped edge or point to give consistent results. With the microcharacter, results were variable, and those obtained by different observers showed rather marked variations. Some such scratch test, however, would be desirable to find out if the alloy contained any constituents that were harder than the steel of the journal. The hardness measured by the Brinell instrument tells nothing about the scratching quality of the different constituents, but, rather, its resistance to deformation. The Brinell test will probably take the place of the compression test in routine testing of bearing alloys, as it is much easier and quicker to carry out.

Transverse Bending Tests. The transverse bending test is of importance in bearings which may be eccentrically loaded. This is

the only case in which part of the bearing metal is under tension. Where such tests have been made in this country it has usually been on the complete bearing to see how it stands up as a whole. In some parts of Germany, however, it has been the practice to make transverse bending tests on bars of the bearing metal itself.

Dynamic Tests. In dynamic tests the material is subjected to varying loads or to impact. While bearing metals are actually subjected to such loads in service, particularly in automotive and railroad service, little has been done to develop methods of testing under these conditions. The results obtained with dynamic tests depend greatly on the machine and type of specimen used, and results of experiments by different methods are not often comparable.

Fatigue Tests. The resistance to rapidly alternating stresses, that is the fatigue resistance, is a property which has been almost entirely neglected in bearing metals. One fatigue test of importance is the fatigue bending test applied to the complete bearing. This is of importance in automotive engine bearings, particularly aircraft engines, in which the linings sometimes fail because of repeated flexing of a back which is not rigid enough.

In fatigue tests, the stresses are usually below the yield point, and the endurance limit is of the order of magnitude of the elastic limit. To give an idea of the resistance of a metal under such conditions, impact fatigue tests are valuable. The specimens are subjected to the impact of a definite weight falling always through the same height. The impact fatigue number is the number of blows required to fracture or to deform by a fixed amount. This information is of importance in railroad service, where flat wheels and consequent poundings are likely to occur. Under such conditions, it is desirable that the bearing shall last until the car can reach a shop. A metal which cracks up or pounds out after a few blows is worthless. The type of specimen and kinetic energy of the blow are entirely arbitrary and each user must choose them to suit his product. This test, however, will be of value in detecting poor material in the course of routine testing.

Notch Bar Tests. The notch-bar impact test, in which a notched specimen is fractured at a single blow, is not so applicable to bearing metals, as they are not exposed to such conditions in service. Most of the bearings have oil grooves, however, which produce some "notch effect". The conditions in bearings would be duplicated

perhaps by repeated impact tests on a notched specimen. The energy of blow being much less than that necessary to fracture the specimen at a single blow.

Wear Tests. Wear tests have not yet been standardized, and such laboratory wear tests as do exist may not always apply to service conditions. However, the recent work at the Bureau of Standards offers much encouragement along these lines.

The mechanism of wear will have to be better understood before standard tests can be established, and "wear" given a standard definition. It is often defined as "any deterioration of the bearing metal due to service, including even cracking." In "wear tests", as generally carried out, the "wear" is a combination of actual wearing away of the metal and distortion. In some tests the bearing metal is rubbed with a steel disk or journal, no nonmetallic abrasive being present, and the loss in weight is determined, this loss being considered a measure of wear.

The recent work on testing of bearing metals at the Bureau of Standards, which has been reported in the proceedings of the American Society for Testing Materials, and in the Journal of Research of the Bureau of Standards in 1928 and 1929, covers a period of several years and indicates the development of a rather satisfactory testing technique with the end in view of finding reasons for the wide variations in the specifications of different railroads and automobile companies, for bearings for similar conditions of service.

The direct result of this work has been to arouse the interest of industrial organizations and technical societies in bearings and bearing metals. While the tests used are not perfect, nevertheless, sufficiently good agreement has been secured to enable some valuable comparisons to be made. This was partly due to the fact that the Amsler wear tests which were employed were made principally on dry specimens which did not heat up even when run at high pressures because the test pieces were in the shape of disks having only a line contact. It was possible under the conditions to run a large number of tests in a reasonable time, and they showed good agreement with different operators at different times.

The literature does not disclose elsewhere a similarly good result. Therefore, it would seem that a distinct advance had been made in the testing of bearing metals by the workers at the Bureau of Standards.

Questions will arise naturally as to the practicability of a dry test, but when it is considered as one of a series of tests and not as the final word, its value cannot be doubted.

With standardized testing technique it becomes possible to determine with some accuracy the effect of small quantities of other metals in the main alloy, thus pointing the way to proper specifications and commercial standards.

Several bearing testing machines have been designed which simulate the actual conditions in a bearing. These operate by hanging the bearing on a revolving shaft and weighting it down by means of a pendulum on which weights are suspended. As the shaft is revolved the friction or the torque is determined by the angle at which the pendulum swings while the shaft is revolving. In such machines the bearings are always lubricated, and the wear of the bearing metal is determined by the loss in weight. At first glance one might think that reliable results could be obtained from such a machine simulating actual conditions. As a matter of fact, results obtained have been very erratic and unreliable, in many instances, due in all probability to the fact that the area of contact was continually changing as the bearing metal wore, and, also, because the lubricant used was a decided factor in the ultimate result. If such a machine is run without lubricant the temperature rise would be very great, but with an Amsler machine which operates with a line contact, pressure several times above that found in practice can be employed easily without any heating of the specimen whatever.

Important results such as those of Dudley on lead bronzes, have been obtained from wear of bearing metals in service, reports of frequency of hot-boxes, etc. Owing to varying conditions of maintenance, however, only rough average values can be obtained from such records. These are valuable in that they give an idea of the reliability of the bearing type and bearing metal in actual service. Information of this character may be obtained from the so-called accelerated service tests in which bearings are run under extreme conditions of load, speed, or temperature, corresponding to conditions of overload or improper maintenance.

Lubrication

For a long time friction was ascribed to unevenness of the

surfaces which touched. Projections interlocked and thus offered a resistance to the relative motion of the surfaces. Nevertheless it has been shown that when these projections have been polished out to almost absolute flatness that the friction is still great enough to cause a quick wearing away of the surfaces. We are, therefore, forced to the assumption that the unevenness of the surfaces cannot be the only cause of friction, and we have to seek in the inner molecular differences themselves for the further cause of friction.

According to the modern scientific conception, friction depends in all cases on an interaction but not on an interlocking of the roughness of the surfaces, but above all on an interlocking of the heterogeneous electromagnetic fields of force which radiate from the surfaces. But if this is the nature of friction, it logically follows further, that a decrease in friction can only be reached by diminishing, in some way or another, this interlocking of the electromagnetic fields of force. This is exactly the task of the lubricant which we bring between the rubbing surfaces.

There are two types of lubrication (1) thick film (complete lubrication), (2) thin film (incomplete lubrication). With complete or thick-film lubrication the lubricating film is so thick that friction takes place entirely within the lubricant at a distance from both rubbing surfaces, outside the reach of the fields of force radiating from the surfaces. We understand that in this case the kind and intensity of the two fields of force is entirely without significance, because the thickness of the lubricating film does not permit an interlocking of the fields of force.

In order to maintain a thick film of any fluid lubricant it is necessary, in general, for the bearing surfaces to be inclined to one another at a slight angle. This permits the formation of a wedge of the lubricant, which tends to separate the bearing surfaces and thus serves to support the load. If the surfaces are parallel or inclined the wrong way this wedging action is lost and the main body of the lubricant escapes.

The load-carrying action of a wedge-shaped oil film is similar to that of the air directly under the wing of an airplane in flight. In each case a fluid pressure is developed on the under side of the inclined surface purely as a consequence of the relative motion. On the upper surface of the airplane wing, and on certain portions of the lubricated bearing surface, negative pressures are also developed,

and it is simply the resultant of all these positive and negative fluid pressures that supports the load.

Besides having the proper inclination of bearing surfaces, a second prerequisite to insure the maintenance of a thick film is obviously that of having an adequate supply of lubricant in immediate contact with the moving surface.

The only property of a fluid lubricant which can appreciably influence its mechanical action in thick film lubrication is its viscosity. We refer to its viscosity at the true temperature of the film, not at the room temperature or at some arbitrary standard temperature. The chemical composition of such a lubricant has no influence on the result except indirectly through changes that may occur in its viscosity.

The nature of the bearing metal can have no direct influence in thick film lubrication provided the oil actually wets the metal.

Thin Film Lubrication. Any machine at the moment of starting or stopping or reversing its direction passes through zero speed and thereby fails to meet one of the requirements for the maintenance of a thick film. Bearing surfaces that are too heavily loaded on small areas cannot support a thick film; gear teeth and cutting tools, for example. Parallel surfaces like the ways of a planer and the rubbing surfaces of a piston and cylinder cannot in general be expected to maintain a thick film, although they may do so momentarily.

During the starting interval such as mentioned above there is a period during which as the speed of the shaft is increased the coefficient of friction becomes progressively less until a minimum point is reached. Beyond this minimum point any increased speed results in an increased coefficient of friction which is a function of the speed of the shaft.

This transition from a decreasing coefficient to an increasing coefficient of friction is the dividing line between two recognized types of lubrication.

During the period of decreasing coefficient of friction thin-film lubrication prevails and the bearing metal and the oiliness of the lubricant play an important part. As the coefficient of friction increases these factors disappear as complete lubrication (known as thick film lubrication) is obtained.

In heavy machinery the effect of the speed is to center the journal in the bearing, so that it is surrounded with quite a uniformly thick lubricating film. In fine mechanisms, where the plane in which

friction takes place is not far enough removed from the rubbing surfaces to be completely outside the influence of their fields of force, the case of so-called incomplete film lubrication is the rule.

Thin film lubrication depends on some unknown factor or group of factors collectively known as the property of *oiliness*.

Oiliness of a lubricant is defined by Hershel as the property which causes two lubricants of like viscosity, at the temperature of the lubricating film, to give different friction when used under the same conditions. Generally speaking, lubricants of high oiliness adhere to the bearing surface with greater force than lubricants of low oiliness. Recent tests of oiliness have disclosed the fact that a lubricant may not have the same oiliness when tested in conjunction with different bearing metals.

With incomplete film lubrication, the lubricant will fulfill its function of decreasing friction so much the better, the better it is in position to neutralize the fields of force radiating from the solid surfaces, and to eliminate them as a factor causing friction. For this purpose, in the first place, it is necessary to maintain a continuous film of oil, be it ever so thin, to cover the solid surfaces and offer sufficient resistance to the tangential forces tending to destroy the films. Moreover, the molecules of the lubricant can have special properties for reducing friction, which further increase the effect of the mere presence of the oil film. The sum of these phenomena which maintain a lubricant film under the influences of solid surfaces, and decrease friction without more fluid friction being caused within the lubricant, is what Cuypers calls the oiliness of the lubricant, i. e. "While with complete film lubrication the viscosity is the factor which causes a decrease in friction, the cause of reduction of friction in the case of incomplete film lubrication is not the viscosity, but the oiliness of the lubricant."

It is the incontrovertible service of Woog to have most thoroughly investigated the factors upon which good oiliness depends, and to have devised a system to accomplish this. It would lead much too far in this place to go further into these most interesting investigations of Woog, but according to him the most important factors which determine oiliness are:

1. The mutual keying of the molecules of the lubricant together, by which we understand the resistance they oppose to pulling apart, in-so-far as it depends on a single molecule's having a hold on its neighboring molecules. For this hold, the size of the molecular

volume is of predominant significance. The greater the molecular volume of a lubricant so much firmer does it hold even the thinnest film together.

2. The form and dimensions of the lubricant molecule: We should not imagine the molecules as symmetrical, spherical shapes, but as less complicated forms showing strong irregularities. An oil film which consists of long molecules which stand close together like the bristles of a brush is much more resistant to the tendency of rotation than is an oil film consisting of molecules more nearly spherical.

3. The presence of specially active centers of energy in certain molecules, which forces them under certain conditions to take on quite different positions: especially effective are such centers of energy when they are located at the end of the molecule. It appears that the durability of the oil film standing in direct contact with the solid surfaces will be so much the greater the firmer the lubricant molecules anchor themselves to the surfaces. And this anchoring will always be especially intensified if the force of attraction due to the metal surfaces themselves is strengthened by a special affinity of the lubricant molecules in which even such active centers of energy orient themselves towards the surfaces.

4. The fourth factor of oiliness of a lubricant is the elastic stiffness of its molecules.

The comparison of results obtained by experiment with practical results is very interesting. In practice it has always been found and maintained that the lubrication action of fatty oils, of animals and plants, is better than for mineral oils. For a long time science which at first regarded the problem of lubrication from a purely physical point of view of fluid friction, has thought this experience must be controverted, as neither the viscosity, which was at first mentioned, nor the surface tension which was later thought to be of significance in regard to the lubricating efficiency of an oil, gave any conclusive results in favor of the fatty oils. The matter appears differently today. The better lubrication efficiency of the fatty oils, always maintained in practice, does not rest on their viscosity nor on their surface tension (there are enough mineral oils which show the same characteristics in these respects) but in their essentially greater oiliness.

All of the factors coming into the question of the formation of high oiliness are present with them to an extreme. They have a

much greater molecular volume than equally viscous mineral oils, their form is markedly long—three times as long as wide—and they possess in contrast to mineral oils, polar active centers of energy which act to anchor their molecules fast to the rubbing surfaces. On these grounds, an adsorbed film of fatty oil is always more resistant than an adsorbed film of mineral oil of similar viscosity and therefore the effective decrease of friction in so-called half fluid friction is always greater with lubrication by fatty oils and more persistent than with lubrication with mineral oils.

The Effect of Bearing Metals on Lubricants

Quite unexplained are the effects of the metal parts on oil. There is no doubt that such effects exist to a considerable extent, and they may be due to the nature of the metal itself, to the manner of its preparation or to the handling of its surface. As regards the metal itself, we know at least this much, that certain metals act catalytically to accelerate the decomposition of fatty oils. This is especially true of copper, zinc, nickel and apparently also antimony and the alloys of these metals. Then it is possible that certain methods of finishing originally inert metals as, for example, iron, may lead to the formation of chemical compounds in the metal which will act especially unfavorably on fatty oils. And, finally, contamination of the surface from handling, as in hardening, polishing, etc., could result in chemical combinations of neatsfoot oil for example, which is an oil that may be regarded as specially favorable in respect to its chemical structure, so that a viscous soapy mass would be formed which would have a braking effect instead of lubricating.

It has been shown that one or two per cent of such fatty compounds as oleic and stearic acid when added to mineral oils, increase the lubricating properties tremendously, so that it is important to study the action of the bearing metals, for example, on such as molecules of stearic acid, with a high molecular value, so that its efficiency in the lubricant may not be destroyed.

Largely because of cost, mineral oil is the common lubricant and it has been shown that the action of one or two per cent of such fatty compounds as oleic and stearic acids increase its lubrication power tremendously, therefore, in order to retain this superior quality it is essential that we study bearing metals so that none will be

used which will react in such a way as to destroy these valuable qualities, thereby decreasing the value of the lubricant.

Therefore, the best lubrication is secured by increasing the durability of the fatty oils, by the elimination of unsuitable bearing metals or such treatments of them as outlined above as have been found by experience to attack the sensitive structure of the fatty oil used.

All of the above would seem to indicate the tremendous importance of the study of bearing metals and their reaction on the lubricant, in order to be able to select those that act more favorably under definite conditions.

The recognition of the nature of so-called incomplete film lubrication is new, as is the significance of oiliness. The conception of oiliness is new and also the search for the factors upon which it depends. New and interesting is finally the fact that in the phenomena which at first glance appear to be purely physical, like the reduction of friction, chemical action should be of such predominant importance.

The information thus far obtained concerning oiliness offers a very rich field for further chemical investigations. Until now the problem of lubrication has been attacked solely from the side of the lubricant. Since, however, in the case of friction in fine mechanisms, the fields of force of the rubbing surfaces play such a considerable role, and these are entirely dependent upon the nature and structure of the surface molecule, we must in the future turn our attention much more than formerly to the material of the rubbing surfaces, and to the affinities between certain groups of molecules in the lubricant and the molecules of the metal surfaces. In this way there is considerable probability of discovering alloys which will be more favorable for orienting certain centers of energy of the lubricant molecules, than many alloys previously used, and thus will better insure an adequate molecular lubricating layer.

In the second place exact investigations of the occurrence or non-occurrence of catalytic action of certain metals on fatty oils might be under certain circumstances of the greatest value for the entire industry of fine mechanics. For example, in the case of certain instruments where the bearing is now made of brass there is always trouble with the oil, whereas if the brass was replaced by tin or tinned metal, a much greater durability of the oil might be attained.

and this greater reliability in service and greater life of the apparatus would permit a sufficient increase in price to pay for the tin or the tinning.

Finally, it would pay to make a general investigation of the influence of the fatty acids on the different metals. The practician is always inclined to understand by the word "acids" something quite dubious and dangerous. As a matter of fact, there is a great difference between saturated and unsaturated fatty acids, between liquid and solid acids, and all acids do not attack all metals to the same degree. Experimental tests as to which acids are of a harmless nature and which are dangerous, on what alloys they act strongly and which they attack less, would be of interest to all who use oil. Such tests would be all the more to be welcomed since in Woog's process the stearic acid is the chief agent for the reduction of friction.

That by and for itself, the free acid present in the lubricant is very effective in reducing the friction, has long been known. For example, in England, Wells and Southcombe proposed in 1920, as a result of their studies, to increase the lubricating value of ordinary mineral oils by the addition of a minute amount of fatty acid.

It would appear, therefore, that there are a number of further tasks which await scientific investigation and explanation, whose solution would be of greater value for every oil user and oil maker than all practical tests on single instruments and single lubricants.

A STUDY OF THE TUNGSTEN-CARBON SYSTEM

By W. P. Sykes

Abstract

This paper describes the results of a systematic study of alloys of tungsten and carbon. A large variety of compositions were examined as formed in the solid and also by melting in a carbon resistor crucible under reasonably close temperature control.

From the data furnished by chemical analyses, microstructures, melting temperatures and X-ray diffraction patterns, a tentative constitutional diagram was constructed covering the tungsten-rich end of the system.

Tungsten appears to dissolve in the solid about 0.05 per cent carbon at 2400 degrees Cent. (4350 degrees Fahr.) The first eutectic melts at about 2475 degrees Cent. (4485 degrees Fahr.) and has a composition close to 1.5 per cent carbon. It appears to consist of the tungsten-rich phase and the compound W_2C (3.16 per cent carbon). This compound melts at 2650 to 2750 degrees Cent. (4800 to 4980 degrees Fahr.)

The second eutectic, melting at about 2525 degrees Cent. (4575 degrees Fahr.) has a carbon content of approximately 4.5 per cent. It consists of the compounds W_2C and WC (6.12 per cent carbon).

The latter phase, WC, begins to decompose at about 2600 degrees Cent. (4710 degrees Fahr.) forming a tungsten-rich liquid and solid carbon apparently in the form of graphite.

SINCE the first description of tungsten carbide was published, by Moissan[1] in 1896, the alloys of tungsten and carbon have been studied by several investigators. A complete bibliography of the subject is included in a recent paper by Gregg and Küttner.[2] In this literature are found references to the compounds W_3C, W_2C, W_3C_2, WC and W_3C_4.

[1] H. Moissan, "Recherches sur le tungstène." *Comptes Rendus* (1896) 123. 13.

[2] J. L. Gregg and C. W. Küttner, "A Metallographic Study of Tungsten Carbide Alloys" Technical Publication No. 184. Institute of Metals Division, American Institute of Mining and Metallurgical Engineers. 1929.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, W. P. Sykes, a member of the society, is metallurgist, Cleveland Wire Works of the General Electric Co. Manuscript received June 13, 1930.

The most extensive investigation of fused alloys of tungsten and carbon is that reported by Ruff and Wunsch[3]. A brief summary of their methods and conclusions is included here for purposes of comparison with the present study.

In the work of Ruff and Wunsch tungsten metal was partially carburized in illuminating gas and then heated in a carbon crucible in a vacuum. The melting of the charge was observed through an opening in the wall of the crucible and the temperature determined with an optical pyrometer. The charges were analyzed before and after melting. Analyses showed that a marked increase in carbon content had occurred during the melting operation.

These authors observed in their conclusions that the apparent melting point of the charge would depend upon the extent of the carburization taking place during the final heating. This would, in turn, be governed by the time of heating, by the temperature and also by the original carbon content of the charge.

From their observations of melting temperatures, microstructures and the results of chemical analyses, they conclude that the lowest carbide is W_3C (2.12 per cent carbon), which melts above 2700 degrees Cent. (4890 degrees Fahr.) They think it probable that W_2C (3.16 per cent carbon) also exists and show a melting point of 2810 degrees Cent. (5090 degrees Fahr.) for this composition. The third carbide WC (6.12 per cent carbon) is described as decomposing at about 2685 degrees Cent. (4865 degrees Fahr.) into graphite and one of the lower carbides.

Three eutectics are mentioned; the first, at about 1.4 per cent carbon, melting near 2690 degrees Cent. (4875 degrees Fahr.) and consisting of tungsten and W_3C. The second is described as a probable metastable ternary eutectic, containing about 2.4 per cent carbon consisting of the three carbides and melting at about 2660 degrees Cent. (4820 degrees Fahr.). The third eutectic, placed at about 3.5 per cent carbon, consists of the WC and probably W_2C and melts at about 2580 degrees Cent. (4675 degrees Fahr.).

They further state that the solubility of carbon in solid tungsten must be less than 0.12 per cent carbon.

The most recent detailed study of the system is reported by Becker[4]. In this investigation tungsten filaments were heated in an

[3]Otto Ruff and Rudolph Wunsch, "Arbeiten im Gebiet hoher Temperaturen, Wolfram und Kohlenstoff." *Zeitung für Anorganische Chemie.* 1914, vol. 82, p. 292.

[4]Karl Becker, "Ueber das System Wolfram—Kohlenstoff," *Zeitschrift für Metallkunde,* December, 1928, p. 437.

atmosphere of hydrogen and benzene in which the partial pressure of carbon was controlled, as well as the temperature of the filament. From the diffraction patterns of a series of filaments so treated Becker affirms the existence of the two carbides W_2C and WC. He suggests, moreover, two forms of W_2C, one being stable only at temperatures above 2400 degrees Cent. (4350 degrees Fahr.).

These results are for the most part in agreement with the findings of Andrews[5], who identified the compounds W_2C and WC by vapor pressure measurements in a study of the reaction between tungsten and napthelene at elevated temperatures.

With the two exceptions noted, all the investigators have worked with alloys of tungsten and carbon produced by melting mixtures of the two elements under a carbon arc or in a carbon crucible. This method naturally leads to melts varying in carbon content from surface to center unless the liquid has been saturated with carbon at the temperature of the melt. Consequently a co-ordination of chemical analyses and microstructures has been, at best, an uncertain method of identifying the phases.

Method and Materials

Because of the relatively high temperatures involved in this system, a resistor crucible of carbon seemed best adapted for melting. The crucible is shown in section on the calibration sheet (Fig. 1). This crucible was inserted between heavy carbon blocks (Fig. 2) in a circuit controlled by a regulating transformer. The temperature-amperage curve for this crucible was drawn from a series of observations made with an optical pyrometer on the surface of the crucible heated in still air. In melting the alloys, the crucible was heated in a partial vacuum (2 to 5 millimeters) within a water-cooled jacket. This practice served mainly to protect the carbon from excessive oxidation and permitted heating periods of 5 minutes or longer, without appreciable change of resistance in the crucible. A partially-melted charge is shown in position in a vertical section of the crucible in Fig. 3.

In order to approximate the temperature at the interior of the crucible under operating conditions, the amperage corresponding to the melting point of pure molybdenum [2620 degrees Cent. (4750

[5] M. R. Andrews, "Production and Characteristics of the Carbides of Tungsten," *Journal of Physical Chemistry*, 1923, vol. 27, p. 270.

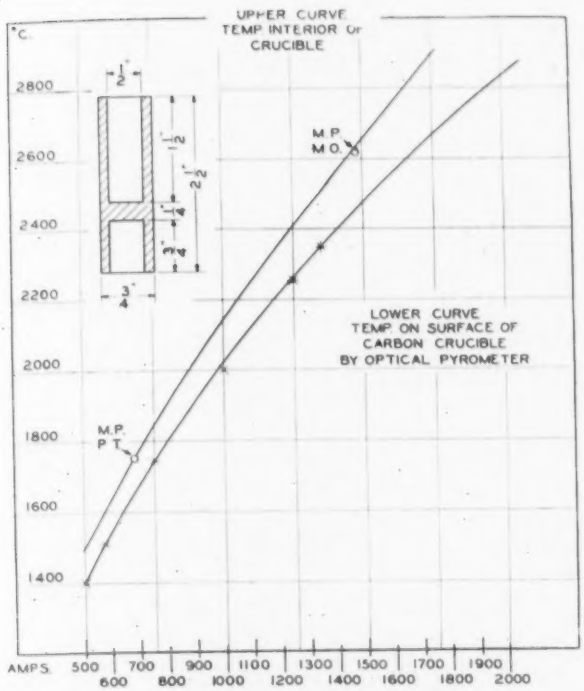


Fig. 1—Temperature-Amperage Calibration Curve for Carbon Crucible.

degrees Fahr.)] was determined within plus-or-minus 25 amperes (at 1475 amperes). This determination was made by heating rods of molybdenum metal in the crucible for 30 seconds at each of a series of current values. The specimen was mounted in a carbon base and the metal extended upward into the hottest portion of the crucible and well out of contact with the inner surface. Between the upper and lower amperages fixing this bracket, (1450-1500 amperes) the metal was completely fused and microscopic examination of the resulting melts showed that the eutectic of molybdenum and carbon was present only in the outer shell.

The melting point of platinum, 1755 degrees Cent. (3190 degrees Fahr.), was likewise placed between the two current values of 675 and 700 amperes. While this method of temperature control is not highly accurate, it, nevertheless, renders possible the duplication of results.

Formation of Carbides

The carbides of tungsten may be readily formed on the surface of a block of pressed tungsten powder by heating in a carburizing

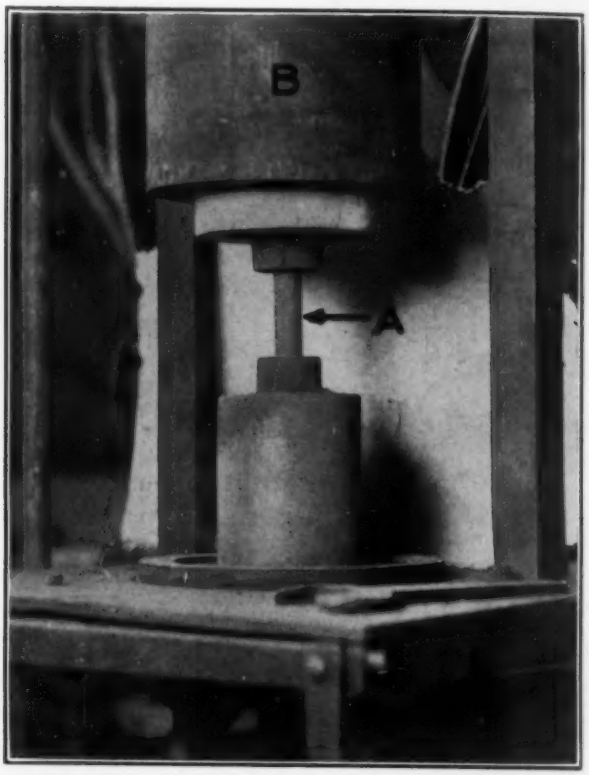


Fig. 2—Resistor Crucible (A) in Position. The Water-Cooled Inclosing Sleeve (B) is Shown in Raised Position.

atmosphere. In Fig. 4 are shown the shells of carbide formed on a block of tungsten by heating at 1500 to 1600 degrees Cent. (2730 to 2910 degrees Fahr.) for 24 hours within a carbon tube surrounded by a hydrogen atmosphere. By this treatment the carbon is apparently supplied to the tungsten in the form of a hydrocarbon resulting from the reaction between the hydrogen and the carbon of the tube at such elevated temperatures.

The fracture of such a block of metal shows two distinct shells. The inner shell has a brownish tint and the outer is gray in color.

These shells are clearly identified by their X-ray diffraction patterns and their reaction to etching reagents. The outer shell is WC; the inner is W_2C.

An extremely sharp line of demarcation exists between the two carbides formed by this method (Fig. 5.). The boundary line between the inner shell of W_2C and the tungsten core is likewise well-defined (Fig. 6).

Tungsten which has been previously heated at about 3000 degrees Cent. (5430 degrees Fahr.) in hydrogen for 10 to 15 minutes, is less rapidly converted to the carbides by the treatment described above. Such material, however, upon heating in the carbon crucible for five minutes at 2400 degrees Cent. (4350 degrees Fahr.) to 2450 degrees Cent. (4440 degrees Fahr.) develops a shell of W_2C

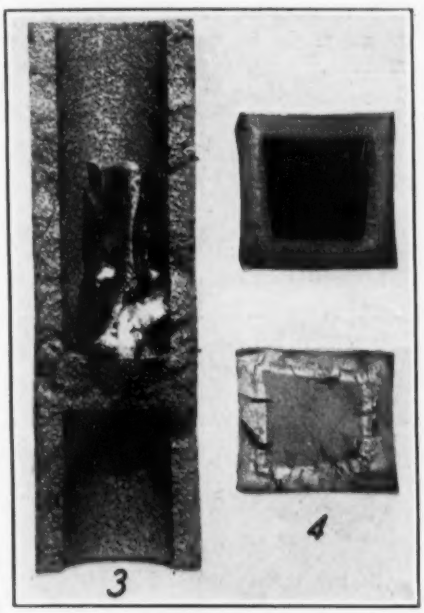


Fig. 3—Partially Fused Charge in Position in Crucible.
Fig. 4—Sections of Briquette of Tungsten Powder After Heating in a Carbon Tube in Hydrogen at 1500 to 1600 degrees Cent. (2730 to 2910 degrees Fahr.) for 24 Hours. Bottom; Fracture. Top; Polished Section. × 2.

to a depth of about .005 inch as shown in Fig. 7. The penetration of carbon into the tungsten core is evidenced by globules and stringers of W_2C lying, for the most part, along the grain boundaries of the tungsten and extending for some distance beyond the inner surface of the W_2C shell. (Figs. 7 and 8).

In the shell of W_2C the tungsten-rich phase is also observed, both as an intergranular network and also in lamellar form within the W_2C grains. Figs. 9 and 10 trace the continuity of this phase from the tungsten-rich core into the adjoining W_2C areas. This lamellar formation decreases in quantity as the field is moved outward from the tungsten-rich core until the W_2C phase alone occupies the entire field. (Fig. 11).

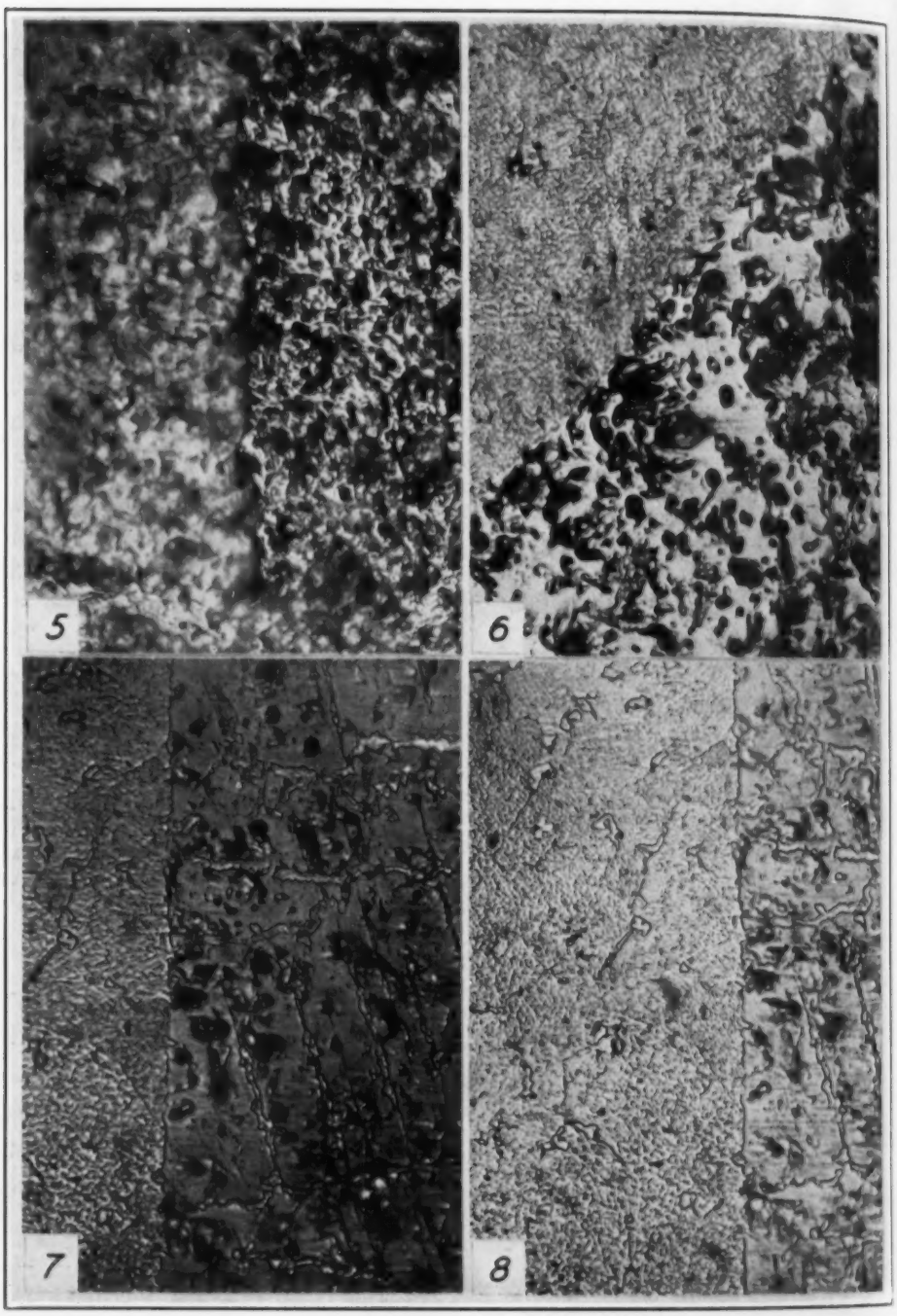


Fig. 5—Boundary Between WC (Right) and W_2C (Left) in Specimen Shown in Fig. 4. The W_2C is Lower and Out of Focus. Etched Alkaline, K_3FeCN_6. × 500

Fig. 6—Same Specimen as in Fig. 5. Border Between W_2C (Right) and Tungsten Core (Left). × 500.

Fig. 7—W_2C on Surface of Tungsten After Heating in Carbon Crucible at 2400 to 2450 Degrees Cent. (4350 to 4440 Degrees Fahr.) for 5 Minutes. Etched Alkaline K_3FeCN_6. × 500.

Fig. 8.—Same Specimen as in Fig. 7. Showing Penetration of W_2C Within the Tungsten Core. × 500.

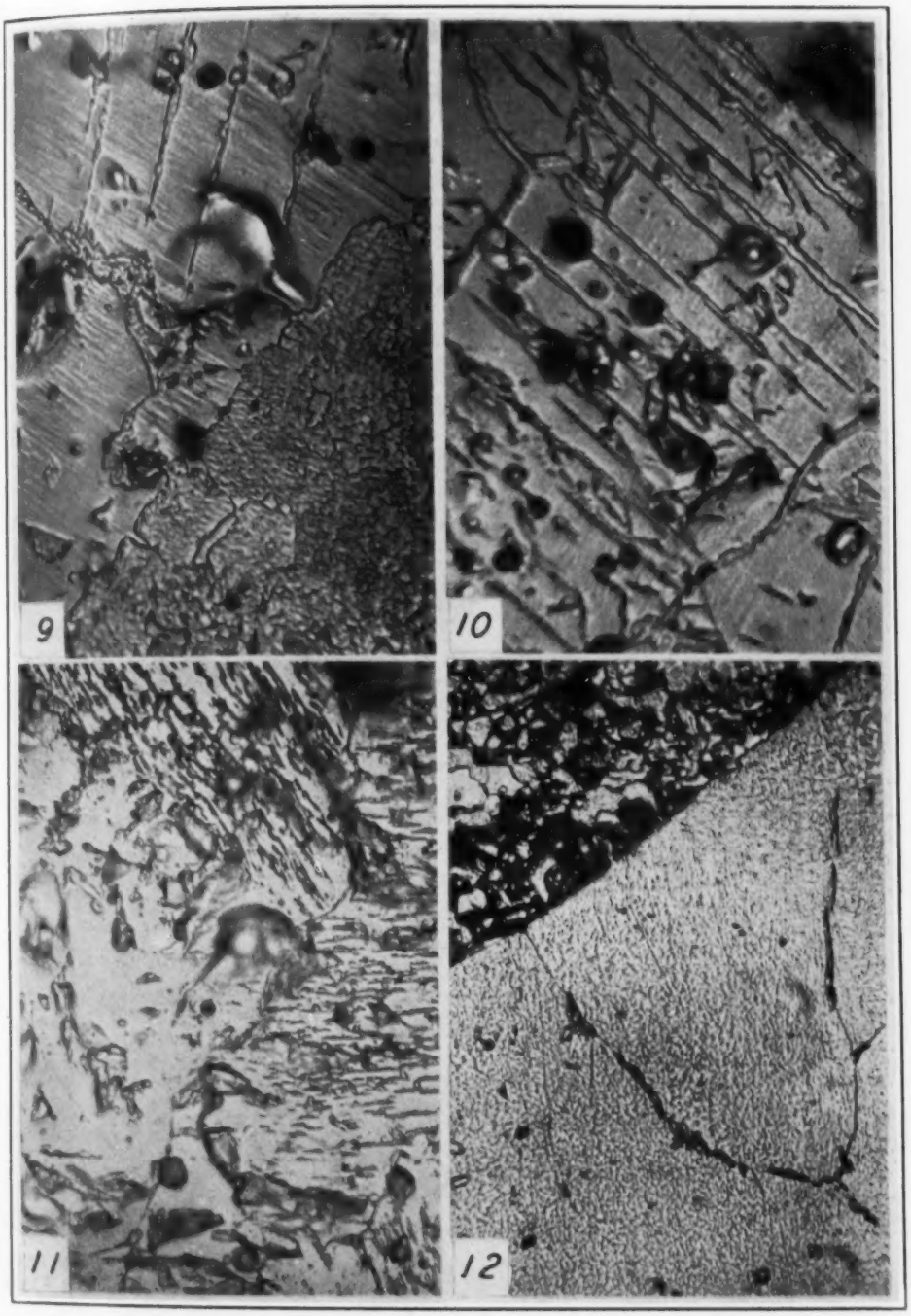


Fig. 9—Junction of W_2C Shell (Left) and Tungsten Core on Tungsten Heated in Carbon Crucible 2400 to 2450 Degrees Cent. (4350 to 4440 Degrees Fahr.) for 5 Minutes and Cooled to 2000 Degrees Cent. (3630 Degrees Fahr.) in About 2 Minutes. Note Continuity of Core and Intergranular Network. × 1000.

Fig. 10—Same Specimen as in Fig. 9, Showing Precipitate of Tungsten-Rich Phase Within the Massive W_2C. × 1000.

Fig. 11—W_2C Intermediate in Position Between the Eutectic W-W_2C to the Right of Field (not shown) and W_2C Seen at the Left. Etched HF+HNO_3. × 500.

Fig. 12—Eutectic W-W_2C at Surface of Carburized Tungsten Specimen Heated in Carbon Crucible at 2500 Degrees Cent. (4530 Degrees Fahr.) for 1 Minute. Etched HF+HNO_3. × 500.

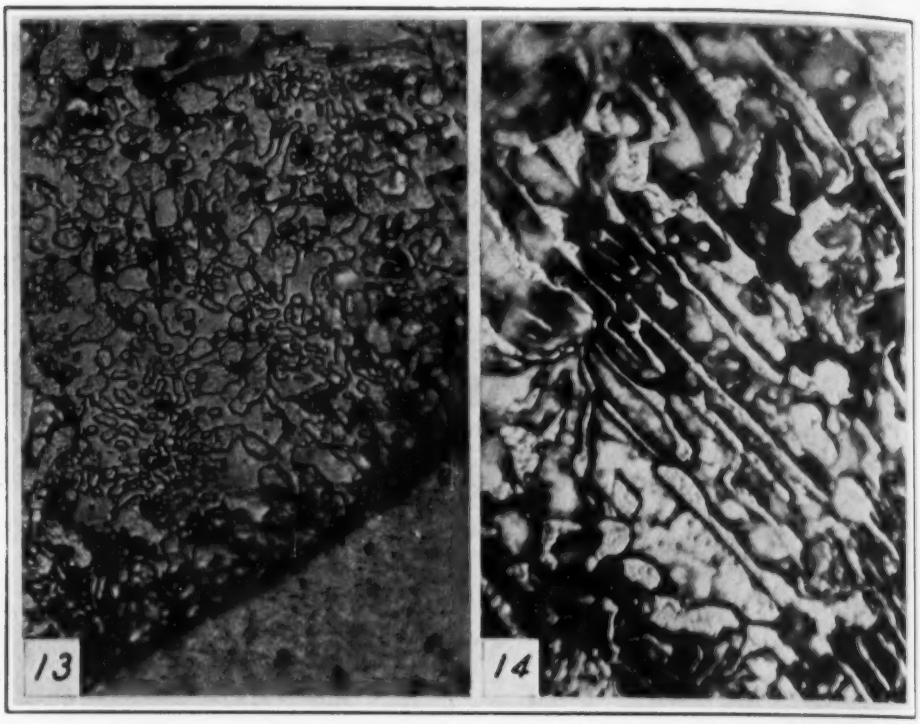


Fig. 13—Same Specimen as in Fig. 12. Showing More of Eutectic Zone. Etched Alkaline K_3FeCN_6. × 500.
Fig. 14—Eutectic of Tungsten-Rich Phase (in relief) and W_2C. Etched $HF+HNO_3$. × 1000.

The occurrence of the tungsten-rich phase within the W_2C is most abundant and massive when the specimen has been cooled from about 2450 degrees Cent. (4440 degrees Fahr.) down to 1800 degrees Cent. (3270 degrees Fahr.) or below at relatively slow rate. By quenching in water as quickly as possible after heating at 2450 degrees Cent. (4440 degrees Fahr.) this formation is largely suppressed. It seems probable, therefore, that the solubility of tungsten in the W_2C phase decreases with temperature. This feature of the microstructure will be mentioned later in connection with melted specimens. When a specimen of tungsten enclosed in such a shell of W_2C is heated to a temperature between 2450 and 2500 degrees Cent. (4440 and 4530 degrees Fahr.) a liquid is formed. This leaves a crevice or series of pits in the polished section between the tungsten-rich core and the massive shell of W_2C. See Figs. 12 and 13.

The liquid formed at 2450 to 2500 degrees Cent. (4440 to 4530 degrees Fahr.) solidifies with a typical eutectic structure as shown in Fig 14.

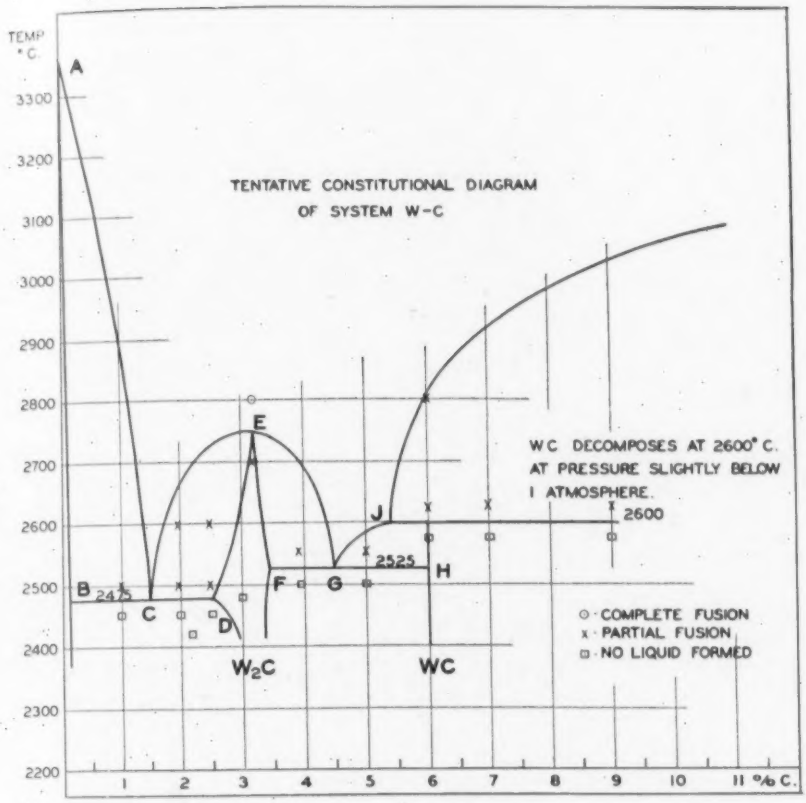


Fig. 15—Constitutional Diagram of Tungsten-Rich Portion of the System Tungsten-Carbon.

Approximation of the Constitutional Diagram

Due to the rapid absorption of carbon from the crucible and atmosphere it is quite impossible to form a melt of uniform composition and structure throughout.

In view of this limitation the microstructures must be interpreted with caution, bearing in mind the original composition of the charge, the time and temperature of treatment and the location within the melt of the area under observation.

A tentative constitutional diagram of the tungsten-carbon system is presented in Fig. 15, based upon the observations which are described in the following pages.

In exploring the range of composition between pure tungsten and W_2C (carbon 3.2 per cent) mixtures were made of tungsten powder and W_2C. The latter constituent was formed from a mixture of 3 per cent lampblack and tungsten powder, heated in a carbon tube in a hydrogen atmosphere at 1500 degrees Cent. (2730

degrees Fahr.) for 2 hours. After screening the mixing, this carbide powder showed by analysis, a carbon content of 3.2 to 3.4 per cent. The theoretical carbon content of W_2C is 3.2 per cent. This material possesses a characteristic X-ray diffraction pattern to be described later, which is identical with that from the inner shell of the carburized tungsten briquette previously mentioned.

The presence of 0.10 per cent carbon added to tungsten as W_2C, is easily detected even after heating the mixture at 1500 to 1600 degrees Cent. (2730 to 2910 degrees Fahr.) for 20 hours. In this case a block of the pressed powders was heated in a short carbon tube in hydrogen. A thin shell of W_2C was formed upon the surface of the specimen but the greater part of the interior was of uniform structure, indicating that the change in composition was limited to the surface regions. (Fig. 16.) Subsequent heating for 2 minutes at 2450 to 2500 degrees Cent. (4440 to 4530 degrees Fahr.) developed a network of liquid shown in Fig. 17. With as little as 0.05 per cent carbon added to the tungsten as W_2C and treated in the manner described above, the presence of a second phase is difficult to detect by microscopic methods. (Fig. 18.) Near the surface of such a specimen, however, carbon absorbed from the atmosphere during the sintering operation results in a network of liquid when the specimen is heated to 2500 degrees Cent. (4530 degrees Fahr.). (Fig. 19.)

The structure of a mixture of tungsten with 0.5 per cent carbon as W_2C heated for some time below the temperature of the W-W_2C eutectic is shown in Fig. 20. The same composition with an additional heating for 2 minutes at 2450 to 2500 degrees Cent. (4440 to 4530 degrees Fahr.) develops an intergranular network of liquid as in Fig. 21.

In order to determine approximately the composition of the W-W_2C eutectic a series of briquetted mixtures containing carbon as (W_2C) in amounts from 0.5 to 3.00 per cent were heated at 2475 to 2500 degrees Cent. (4485 to 4530 degrees Fahr.) for 30 seconds. It was observed that under these conditions the compositions lying between 1.4 and 1.6 per cent carbon were most completely fused. The presence of excess tungsten or excess carbon in the mixture decreased the fusibility to a remarkable extent. The effect of carbon in excess of the eutectic requirement is strikingly shown by the photograph of the melt in Fig. 22. This specimen, originally a mixture of tungsten and 1.5 per cent carbon (as W_2C),

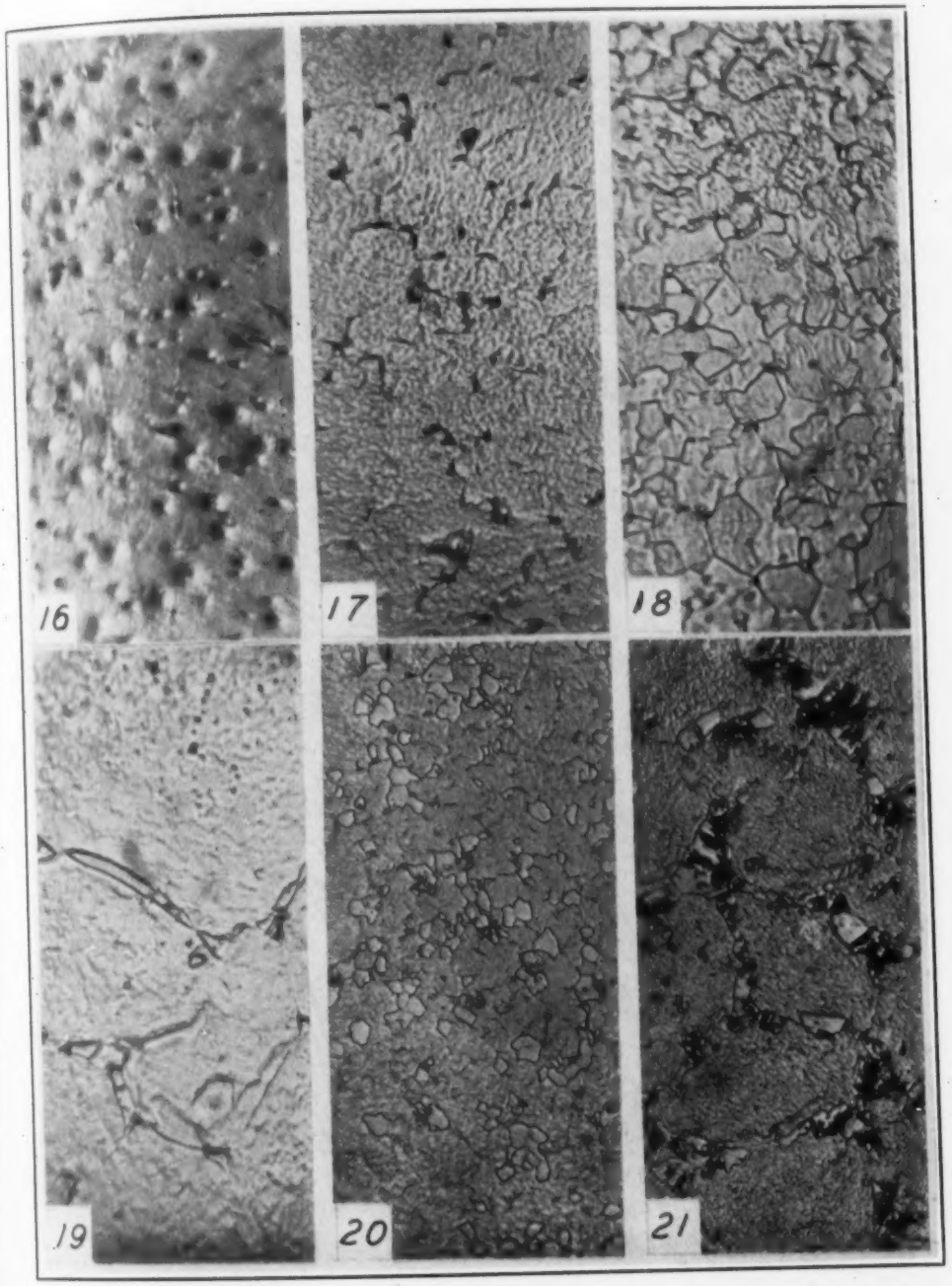


Fig. 16—Tungsten Containing 0.10 Per Cent Carbon as W_2C. Interior of Specimen Heated at 1500 to 1600 Degrees Cent. (2730 to 2910 Degrees Fahr.) for 15 Hours, in Carbon Tube. Electrolytic Etch in NaOH Solution. × 1000.

Fig. 17—Same Specimen as in Fig. 16. Subsequentiy Heated 2 Minutes at 2450 to 2500 Degrees Cent. (4440 to 4530 Degrees Fahr.). × 1000.

Fig. 18—Interior of Specimen of Tungsten Mixed with 0.05 Per Cent Carbon as W_2C. Heated at 1500 to 1600 Degrees Cent. (2730 to 2910 Degrees Fahr.) for 10 Hours then at 2450 to 2500 Degrees Cent. (4440 to 4530 Degrees Fahr.) for 5 Minutes. Etched Electrolytically in NaOH Solution. × 1000.

Fig. 19—Same Specimen Shown in Fig. 18. Field Near Surface, Showing Increase in Carbon Content. × 1000.

Fig. 20—Tungsten Mixed with 0.5 Per Cent Carbon as W_2C. Heated at 1500 to 1600 Degrees Cent. (2730 to 2910 Degrees Fahr.) for 15 Hours in Carbon Tube, then at 2400 to 2450 Degrees Cent. (4350 to 4440 Degrees Fahr.) for 5 Minutes. Etched $HF+HNO_3$. × 500.

Fig. 21—Same Specimen as in Fig. 20 After Subsequent Heating at 2450 to 2500 Degrees Cent. (4440 to 4530 Degrees Fahr.) for 2 Minutes. Etched $HF+HNO_3$. × 500.

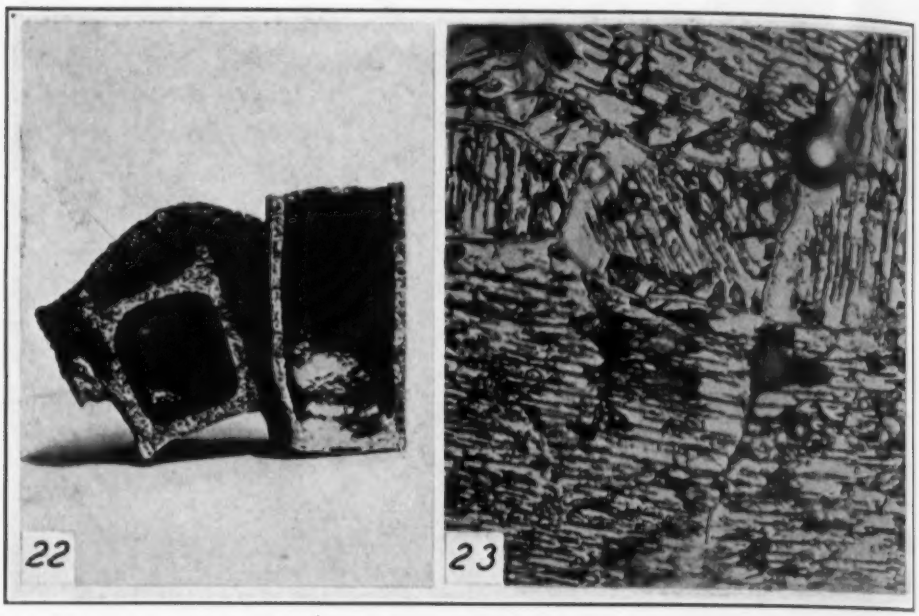


Fig. 22—Mixture of Tungsten and 1.5 Per Cent Carbon as W_2C. Surface Carburized and interior then Melted at 2500 to 2550 Degrees Cent. (4530 to 4620 Degrees Fahr.) as Described in Text. Lower Portion of Charge Shown at Left. × 2½.

Fig. 23—W_2C from Melt of Tungsten-Rich Charge, Showing Intergranular Network and Striations of Tungsten-Rich Phase. Etched $HF+HNO_3$. × 500.

was first heated for 5 hours at 1550 to 1600 degrees Cent. (2820 to 2910 degrees Fahr.) in a carbon tube surrounded by a hydrogen atmosphere. This treatment produced the carbon-rich shell as previously described. The block was then broken in half and one-half placed in a carbon crucible with the fractured surface as a base. Upon heating to 2500 to 2550 degrees Cent. (4530 to 4620 degrees Fahr.) for 30 seconds the interior of the block fused completely and the liquid collected in the bottom of the crucible. This portion of the charge is seen at the left, in Fig. 22. At the right is a longitudinal section of the unmelted shell of the carbon-rich material.

Such surface carburization proceeds very rapidly in the carbon crucible at temperatures above 2000 degrees Cent. (3630 degrees Fahr.). Consequently the exterior regions of melts or partial melts orginally containing less than about 1.5 per cent carbon are found to contain massive W_2C, increasing in amount as the time of heating is prolonged.

In melts of mixtures originally containing between 1.5 and 2.5 per cent carbon, W_2C occurs in large grains bounded and laminated by the tungsten-rich phase as previously described. This structure, Fig. 23, is always found bordered on the one hand by

the W-W_2C eutectic and on the other by the unbroken W_2C phase.

As the carbon content increases above 2.5 per cent the formation of liquid at 2450 to 2500 degrees Cent. (4440 to 4530 degrees Fahr.) appears to cease. Mixtures containing 3.0 to 3.2 per cent carbon can be heated to well above 2600 degrees Cent. (4710 degrees Fahr.) for periods of from 1 to 2 minutes without the formation of any liquid.

Under such conditions, however, the carbon content at the surface increases rapidly and when it has exceeded that of W_2C

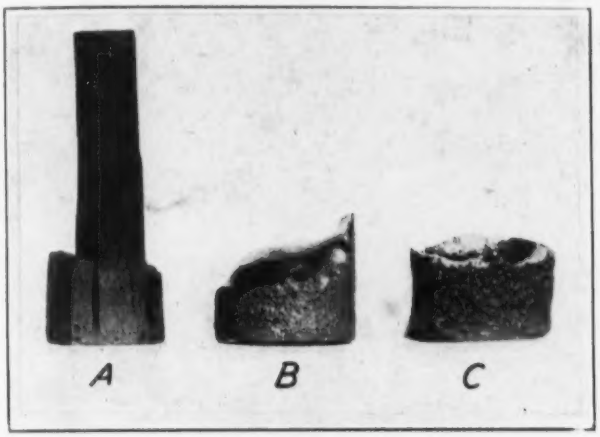


Fig. 24—Speciment of W_2C (Carbon 3.2 Per Cent) Formed as Described in Text. Then Heated in Carbon Crucible.
A. 2650 Degrees Cent. (4800 Degrees Fahr.) for 1 Minute.
B. 2700 Degrees Cent. (4890 Degrees Fahr.) for 15 Seconds.
C. 2800 Degrees Cent. (5070 Degrees Fahr.) for 15 Seconds.

(3.2 per cent) at temperatures somewhat above 2500 degrees Cent. (4530 degrees Fahr.) a liquid forms upon the surface of the charge which appears to be the W_2C-WC eutectic. The true melting point of W_2C is therefore difficult to fix closely by this method. It probably lies, however, between 2650 and 2750 degrees Cent. (4800 and 4980 degrees Fahr.).

In Fig. 24 are shown three charges orginally of W_2C composition (3.2 per cent carbon). It will be noted that no liquid has been formed after heating at 2650 degrees Cent. (4800 degrees Fahr.) for 1 minute. Considerable fusion has occurred, however, at 2700 degrees Cent. (4890 degrees Fahr.) even in 15 seconds. Examination of the second specimen showed the presence of the W_2C-WC eutectic in the outer portions of the melt. A considerable

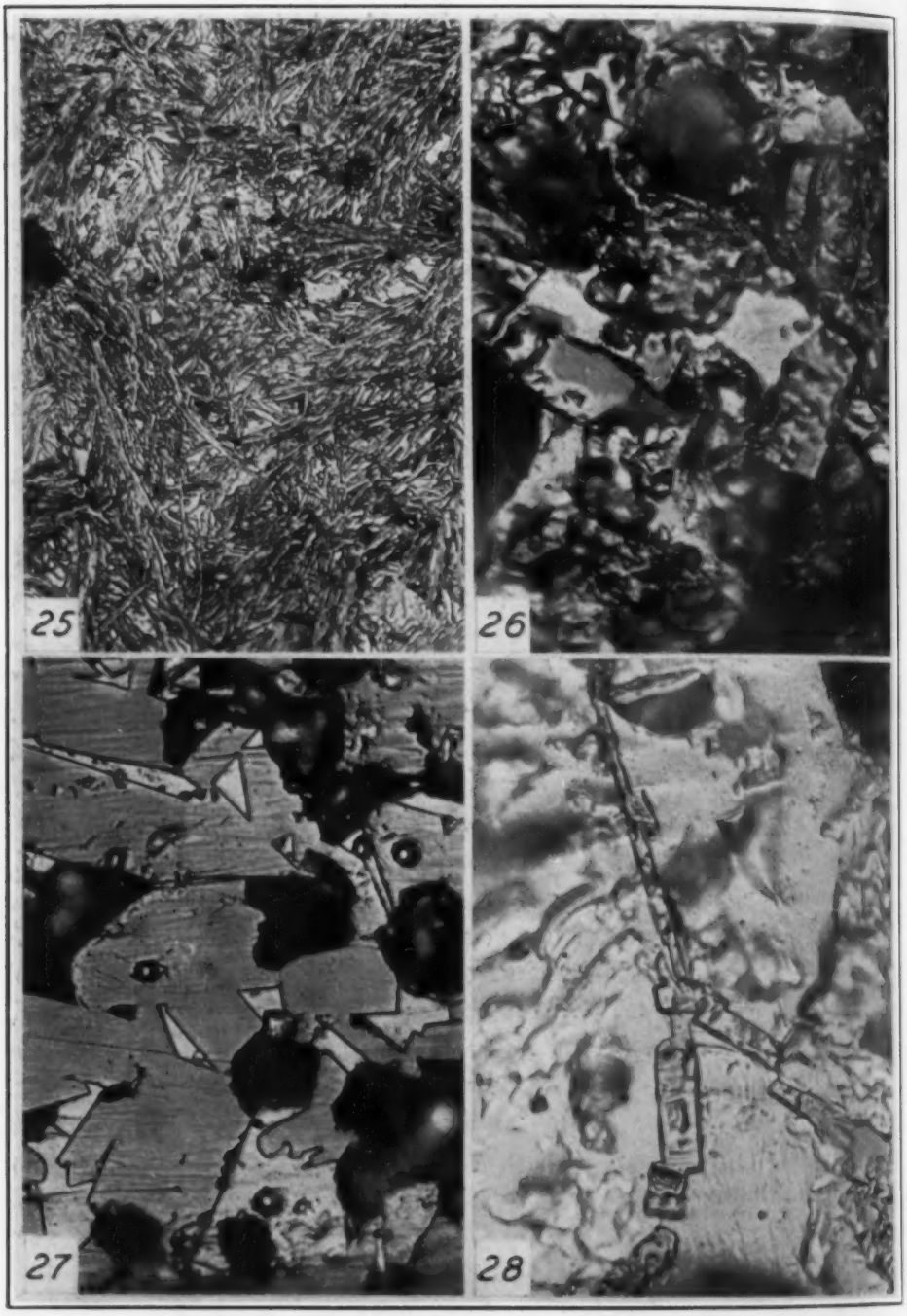


Fig. 25—Eutectic of W_2C-WC in Melt of Mixture of W_2C and WC for Carbon Content of 4.0 Per Cent. Heated 30 Seconds at 2650 to 2700 Degrees Cent. × 500.

Fig. 26—Mixture of W_2C and WC. Carbon Content 4.4 Per Cent by Analysis. Heated 2300 Degrees Cent., 10 Minutes; Heated 2500 Degrees Cent., 2 Minutes; Cooled to 1800 Degrees Cent., in 5 Minutes. × 1000.

Fig. 27—Mixture of W_2C and WC. Carbon Content 5.4 Per Cent by Analysis. Heated 2300 Degrees Cent., 10 Minutes; Heated 2500 Degrees Cent., 2 Minutes; Cooled to 1800 Degrees Cent. in 5 Minutes. W_2C is Lighter Constituent. × 1000.

Fig. 28—Tungsten Containing 3.4 Per Cent Carbon, Previously Formed at 1500 to 1600 Degrees Cent. (2730 to 2910 Degrees Fahr.) as Described in Text. Then Heated at 2300 Degrees Cent. (4170 Degrees Fahr.) for 10 Minutes, 2550 Degrees Cent. (4620 Degrees Fahr.) for 1 Minute and Cooled to 1800 Degrees Cent. (3270 Degrees Fahr.) in 3 Minutes. Field in interior of Specimen. Note the Network of Carbon-Rich Liquid Formed at 2500 Degrees Cent. All Specimens Etched Alkaline K_3FeCN_6. × 1000.

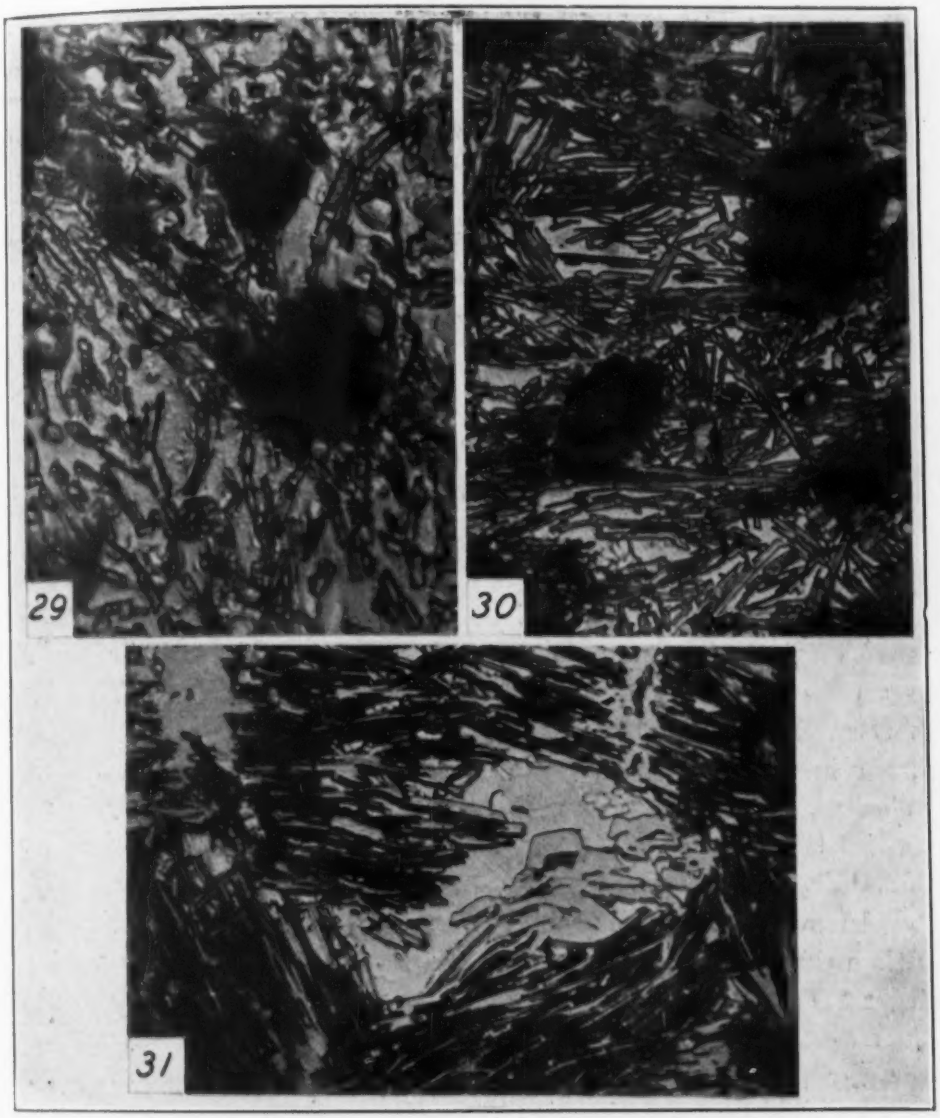


Fig. 29—Same Specimen as in Fig. 28. Field Near Surface in Region of Partial Fusion Due to Absorption of Carbon from Atmosphere. × 1000.

Fig. 30—Eutectic of W_2C and WC in Charge Containing 4.4 Per Cent Carbon (as Mixture of the Two Carbides). Melted by Heating for 30 Seconds at 2500 to 2575 Degrees Cent. (4620 to 4665 Degrees Fahr.).

Fig. 31—Eutectic W_2C and WC with Excess of W_2C (Lighter Constituent). Formed by Partial Fusion of W_2C at 2800 Degrees Cent. (5070 Degrees Fahr.). Etched Alkaline K_3FeCN_6. × 1000.

amount of the W_2C phase remained within, which appeared to have been partly molten.

Proceeding as before, for compositions between W_2C (3.2 per cent carbon) and WC (6.1 per cent carbon) a series of mixtures were made of these two phases as powders pressed into briquettes.

These mixtures begin to melt at a temperature slightly below 2550 degrees Cent. (4620 degrees Fahr.) and fusion goes on most rapidly in the composition range of 4.4 to 4.6 per cent carbon. The structure of the solidified melts of this composition is shown in Fig. 25. This structure is characteristic of material in the composition range between W_2C and WC which has solidified from the liquid state. The needle-like formation can be identified as the WC phase, and is more massive the slower the rate of cooling from 2550 degrees Cent. (4620 degrees Fahr.) or above.

A mixture of the two carbides heated for some time below the solidus or eutectic temperature is shown in Fig. 26. This highly-porous texture is characteristic of these mixtures in which no liquid has been formed. The etch with alkaline K_3FeCN_6 gives the W_2C phase a golden-yellow tint (lighter in the micrograph). The WC phase is distinguished by its gray color (darker in the micrograph). The characteristic colors are more strikingly shown in Fig. 27 in which the WC phase predominates in quantity.

The first indication of a carbon-rich liquid is seen in Fig. 28 as a gray network between the massive grains of W_2C. This field is from the interior of a specimen of 3.4 per cent carbon, slightly richer in carbon than the theoretical compound W_2C. As the title indicates, it was heated to a maximum temperature of 2550 degrees Cent. (4620 degrees Fahr.) and slowly cooled to 1800 degrees Cent. (3270 degrees Fahr.).

In such a composition the outer portions become enriched in carbon during the preliminary heating at 2500 degrees Cent. (4530 degrees Fahr.) A subsequent rise in temperature to 2550 degrees Cent. (4620 degrees Fahr.) results in the formation of a visible quantity of liquid at the surface. In Fig. 29 is seen the structure of such field of local fusion. This is near the surface of the same specimen as in Fig. 28.

A more perfect development of this eutectic structure is to be found in the mixture containing 4.4 per cent carbon which had been fused by heating for 30 seconds at 2550 to 2575 degrees Cent. (4620 to 4665 degrees Fahr.). (Fig. 30.)

This eutectic accompanying a considerable excess of W_2C (the lighter phase) is seen in Fig. 31. In this case, the charge before melting was of W_2C composition, but upon heating at 2800 degrees Cent. (5070 degrees Fahr.) for 30 seconds absorbed sufficient carbon to form a considerable amount of liquid as the W_2C-WC eutectic.

When the carbon content exceeds about 5.5 per cent the certain formation of liquid at 2550 degrees Cent. (4620 degrees Fahr.) is detected with difficulty. Fusion sets in, however, very distinctly at 2600 to 2650 degrees Cent. (4710 to 4800 degrees Fahr.) in the range of composition between 5.5 and 12 per cent carbon as will be seen from the photographs in Figs. 32 and 33.

The liquid formed in such cases freezes with the needle-like structure (Fig. 30) typical of the W_2C-WC eutectic. A charge

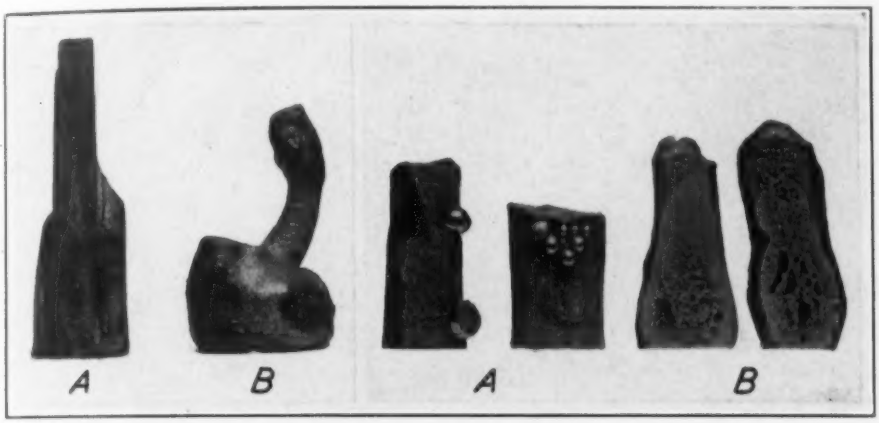


Fig. 32—(Left) Tungsten plus 5.5 Per Cent Carbon, Heated in Carbon Crucible.
A. 2600 Degrees Cent. (4710 Degrees Fahr.) for 2 Minutes.
B. 2650 Degrees Cent. (4800 Degrees Fahr.) for 2 Minutes.

Fig. 33—(Right) Tungsten Plus 6.3 Per Cent Carbon Previously Treated to Form WC (Carbon 6.1 Per Cent). Then Heated in Carbon Crucible.
A. 2600-2625 Degrees Cent. (4735 Degrees Fahr.) — 5 Minutes.
B. 2625-2650 Degrees Cent. (4800 Degrees Fahr.) — 2 Minutes.

containing orginally 5.5 to 6.0 per cent carbon heated at 2650 to 2700 degrees Cent. (4800 to 4890 degrees Fahr.) for a short time (1 to 2 minutes) freezes with a structure shown in Fig. 34. One significant feature of this structure is the crystal of WC with a dark central core. This would suggest that during cooling the first material to freeze is richer in carbon than WC, as perhaps a higher carbide or graphite. The heavy shell of WC surrounding the carbon-rich core and forming the main part of the mass may reasonably have resulted from a reaction between the core and the tungsten-rich liquid surrounding it as the melt reached the peritectic temperature [about 2600 degrees Cent. (4710 degrees Fahr.)] in cooling.

Other crystals of WC of intermediate size and showing no core, are possibly primary crystals which have separated from the liquid at temperatures between 2600 Cent. (4710 degrees Fahr.) and the eutectic temperatures of about 2525 degrees Cent (4575

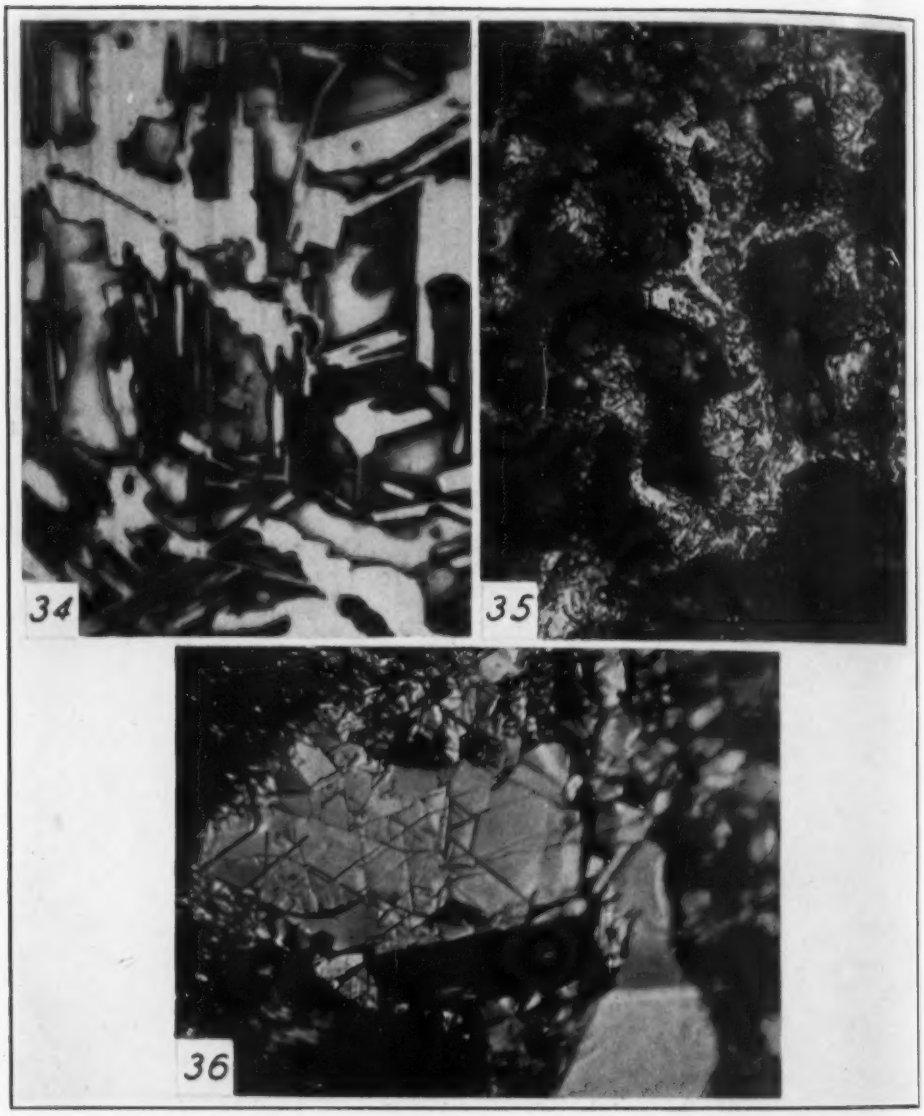


Fig. 34—Tungsten Plus 5.5 Per Cent Carbon. Partially Fused by Heating at 2650 to 2700 Degrees Cent. (4800 to 4890 Degrees Fahr.) for 1 Minute. WC Present in Solidified Melt, as (1) Cored Crystals, (2) Primary Crystals (3) Needle-Like Formation in Eutectic. Etched $HF+HNO_3$. × 2000.

Fig. 35—Tungsten Plus 10.5 Per Cent Carbon. Previously Treated at 1500 to 1600 Degrees Cent. (2730 to 2910 Degrees Fahr.) then Heated at 2800 Degrees Cent. (5070 Degrees Fahr.) for 3 Minutes. Note Network of Fused Material. × 500.

Fig. 36—Surface of Black, Lustrous Crystals occurring in fusions of Tungsten and Carbon Formed at 2700 to 2800 Degrees Cent. (4890 to 5070 Degrees Fahr.) × 25.

degrees Fahr.). At the latter temperature the remaining liquid of approximately 4.5 per cent carbon content has solidified in the form of the W_2C-WC eutectic characterized by the needles of WC in a ground mass of W_2C.

Mixtures of tungsten with 8, 10, and 12 per cent of carbon, added both as lampblack and as graphite, were heated in carbon containers at 1500 to 1600 degrees Cent. (2730 to 2910 degrees Fahr.) in the usual way to form a possible higher carbide. Analyses of the mixtures for total carbon after this treatment showed slight increases in carbon content of 0.1 to 0.2 per cent over the intended composition.

Briquettes of these mixtures upon heating in the carbon crucible showed the formation of liquid first at a temperature just above 2600 degrees Cent. (4710 degrees Fahr.). After heating these higher carbon mixtures at 2800 to 3000 degrees Cent. (5070 to 5430 degrees Fahr.) for 5 minutes, the resulting mass might be best described as a hard clinker of spongy texture, the surface of which is covered by a black lustrous film resembling graphite. In a polished section the liquid network incloses blue-black masses of a solid material which likewise resembles graphite. (Fig. 35.)

The liquid formed in such cases solidifies with the structure typical of the W_2C-WC eutectic. After a few minutes' heating at 2700 to 2800 degrees Cent. (4890 to 5070 degrees Fahr.) considerable liquid has collected in and about the lower portion of the charge. Several analyses were obtained upon material thus enriched in the liquid. In all cases the carbon content was considerably lower than in the upper portions of the charge containing more of the solid constituent. In the portions of the charge remaining upright the carbon content was always somewhat higher than in the original mixture.

Analyses of the fused material formed by heating WC (carbon 6.2 per cent) at 2650 to 2675 degrees Cent. (4800 to 4845 degrees Fahr). for 5 minutes, showed in three instances carbon content of 4.8, 5.2, and 5.5 per cent. Since it is almost impossible to completely free the fused material from the unfused, and since also the surface of the melt is noticeably impregnated with carbon, these analyses serve only as an indication.

These high-temperature melts of originally low carbon mixtures usually contain imbedded in the eutectic matrix, massive black crystals of high luster, Fig. 36. By crushing such a melt and digesting in hydrofluoric acid, the large crystals can be isolated for the most part from the accompanying tungsten-rich material. During the digestion, a considerable quantity of graphite-like mate-

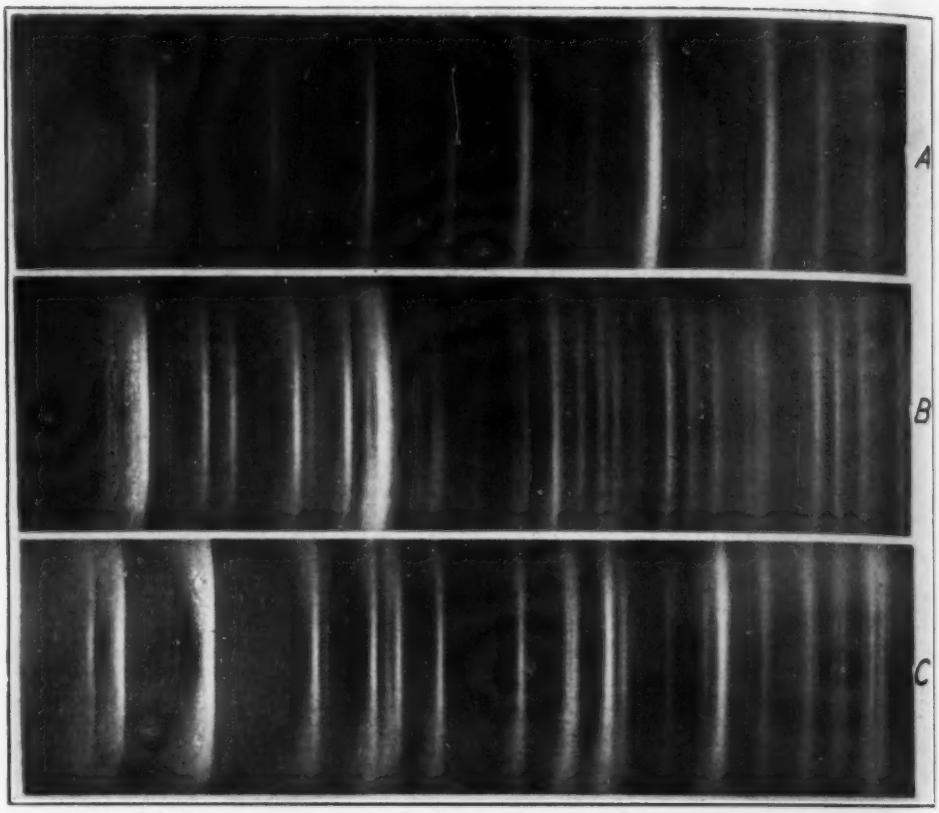


Fig. 37—(A) Tungsten from Interior of Carburized Briquette Shown in Fig. 4.
(B) W_2C (Carbon 3.3 Per Cent). Formed from Mixture of Tungsten powder and Lampblack Heated 2 Hours at 1500 to 1600 Degrees Cent. (2730 to 2910 Degrees Fahr.) in Carbon Tube.
(C) WC (Carbon 6.2 Per Cent). Formed from Mixture of Tungsten Powder and Lampblack and Treated as (B).

rial forms on the surface of the acid and the sides of the platinum container.

The carbon contents of two residues from such digestions were 5.65 and 5.9 per cent, thus approaching the composition of WC. It seems likely that the crystals in question are those of WC associated with graphite.

X-Ray Diffraction Patterns

The diffraction patterns encountered in the many specimens examined are plainly those characteristic of W, W_2C, WC and graphite.

In Fig. 37 are shown the first three of these patterns with a description of the materials from which they were derived.

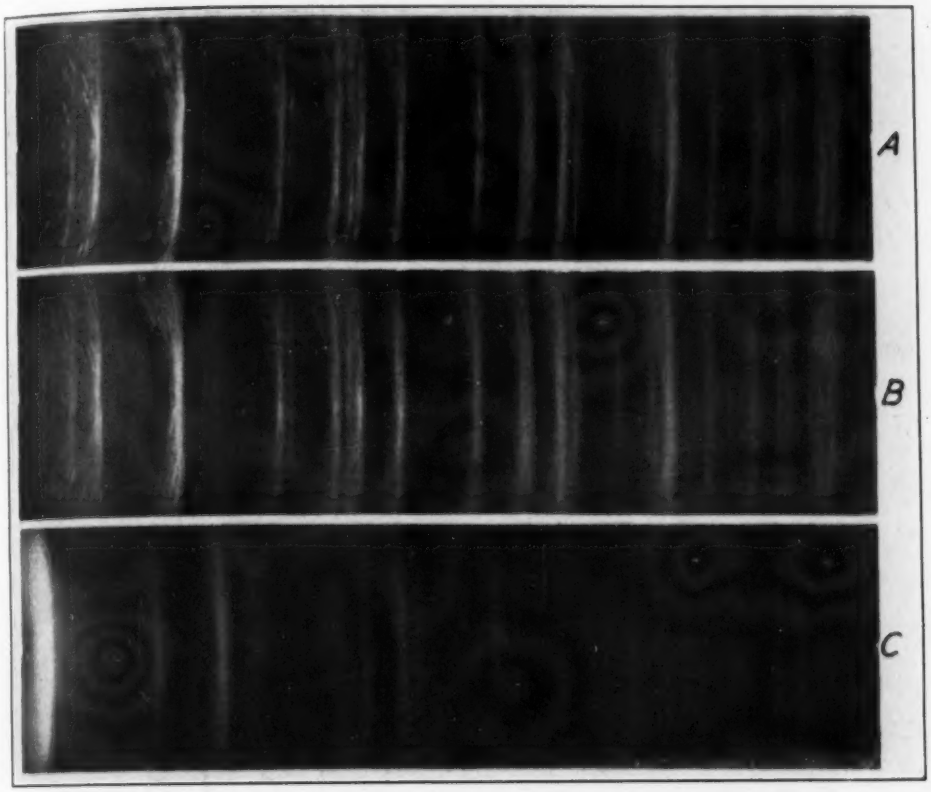


Fig. 38—(A) WC (Carbon 6.3 Per Cent). Material Described in Fig. 37 (C) Subsequently Heated for 10 Minutes at 2450 to 2500 Degrees Cent. (4440 to 4530 Degrees Fahr.) in Carbon Crucible.
(B) Tungsten Plus 12.2 Per Cent Carbon Formed from Mixture of Tungsten and Graphite by Heating at 1500 to 1600 Degrees Cent. (2730 to 2910 Degrees Fahr.) in Carbon Tube for 12 Hours, then Heated at 2450 to 2500 Degrees Cent. (4440 to 4530 Degrees Fahr.) for 10 Minutes.
(C) Pattern from Graphite.

In the composition range between pure tungsten and W_2C (carbon 3.2 per cent) no patterns other than those of W and W_2C were observed. In fused mixtures of W_2C and WC, the W_2C lines ordinarily predominate. No change is noted in the pattern of W_2C after heating at 2500 degrees Cent. (4530 degrees Fahr.) for 2 minutes.

From a partial melt of WC formed at 2650 to 2675 degrees Cent. (4800 to 4845 degrees Fahr.) the pattern is mainly that of W_2C. This doubtless is due to the liquid approaching the W_2C-WC eutectic composition which resulted from the decomposition of WC at the elevated temperature. After such a specimen is reheated at 1500 to 1600 degrees Cent. (2730 to 2910 degrees Fahr.) in a carbon tube for several hours the pattern from the interior of the mass is that of WC alone. This indicates that the subsequent

heating at 1500 to 1600 degrees Cent. (2730 to 2910 degrees Fahr.) has resulted in re-forming the WC phase.

In Fig. 38-B is shown the pattern from a specimen of 12.2 per cent carbon heated to 2500 degrees Cent. (4530 degrees Fahr.). With the exception of an additional faint line at the extreme left this appears to be identical with the accompanying pattern of WC (Fig. 38-A) treated correspondingly.

A pattern of graphite is seen below (Fig. 38-C), which shows an intense line corresponding in position with the additional line in pattern of the 12.2 per cent carbon specimen.

This questionable line occurs also in the patterns from partial fusions at 2800 to 3000 degrees Cent. (5070 to 5430 degrees Fahr.) of charges containing 8 to 12 per cent carbon.

Compositions corresponding to the previously suggested phases, W_3C, W_3C_2, and W_3C_4 were carefully prepared and treated for the purpose of detecting any of these possible constitutents both by the microscope or from the diffraction patterns. No indications of the existence of such phases were observed.

Preparation of Specimens for Examination of Microstructure

The methods used in polishing and etching micro-specimens of tungsten carbide, have been fully described in a recent paper by Dr. S. L. Hoyt.[6]

This procedure was modified only in the case of melts and unfused mixtures containing less than 3 or 4 per cent carbon. Such compositions are too soft to polish well with boron carbide and diamond dust. They are, however, easily prepared by polishing with grain alundum followed by two grades of levigated alumina suspended in water and applied to a broadcloth wheel.

Generally speaking, the most effective etching reagent for this system of alloys is alkaline K_3FeCN_6. When tungsten and W_2C are present together, this reagent attacks the tungsten-rich phase more rapidly than the W_2C, developing the grain boundaries in the former and tinting the W_2C yellow or brown. When W_2C and WC exist together, the contrast between the yellow W_2C and the gray WC is most marked. 25 per cent hydrofluoric acid in nitric acid is a very active reagent, useful in dissolving the W_2C phase which it

[6]Samuel L. Hoyt, "Preparation of Microsections of Tungsten Carbide." Transactions, American Society for Steel Treating. Vol. XVII, No. 1, p. 54.

attacks more readily than either tungsten or WC. This etchant is, however, difficult to control and in porous material the residual hydrofluoric acid, even after careful washing of the specimen, etches the objective lens of the microscope.

ACKNOWLEDGMENTS

To A. B. Gladding of the Cleveland Wire Works, is due all credit for the many X-ray spectrograms involved in this investigation. The author is indebted also to Dr. S. L. Hoyt for his helpful criticism and pertinent suggestions.

DISCUSSION

Written Discussion: By Marcus A. Grossmann, director, Republic Research Laboratory, Republic Steel Corp., Massillon, Ohio.

We are already accustomed to hearing superlatives used in describing Mr. Sykes' work, and I can only add my appreciation to the general commendation.

It is always easy to suggest further work, but Mr. Sykes has only himself to blame if we all look to him to give us further insight into the tungsten alloys. After his earlier work on the iron-tungsten system, followed by the present work on the tungsten-carbon system, we naturally look to him to tell us about the iron-tungsten-carbon alloys. Certainly when he has determined the two compounds WC and W_2C, differentiated clearly as they are in his Fig. 37 by the diffraction patterns, we can hope that he will illuminate the ternary system. Thus we look for evidence locating in the scheme of things the compound Fe_4W_2C found by Westgren and co-workers, a compound so prominent in high speed steels. And we look to him also for information on the range of low-carbon alloys in which austenite is associated with ferrite.

E. C. BAIN:[1] It seems to me that it may not be out of place for some one to point out the enormous difficulties that Mr. Sykes apparently has no fear of in working with this particular system. In the first place, temperatures are all above that at which we are accustomed to work; in the second place he is dealing with compounds that decompose in a way which is not customary in the materials with which we work; in the third place the materials seem to have such sluggishness that he has constantly to check his own work to see that equilibrium has come; and lastly that under the conditions forced by the high temperatures his very composition changes while he heat treats his alloys. And yet by keeping the viewpoint that he has in his early investigations constantly before him he has been able to sort out the essentials from the nonessentials and give us a dia-

[1]Research Laboratory, U. S. Steel Corp., Kearny, N. J.

gram which, I make bold to predict, will not soon be revised. When Mr. Sykes presented the equilibrium diagrams of iron-tungsten and iron-molybdenum, I recall that I said that there were two diagrams which would not need revision. I am going to make the same prediction about this equilibrium diagram.

S. L. Hoyt:[2] It may be of some interest to point out the connection between such an investigation as this and the causes which gave rise to it. Cemented tungsten carbide is fairly well known by this time, but it is quite a recent metallurgical product. We have here simply the reactions of the metallurgist to new problems which are advanced from time to time. From the comments which have already been made and from the work which Mr. Sykes has done in the past, it is not at all strange that he should be looked to to take up such a study as this.

Mr. Bain has pointed out some of the difficulties which I had in mind bringing up. However, that will not be necessary now. Mr. Sykes shows great fortitude, but I must add parenthetically, he derives a great amount of pleasure from tackling just such problems as these which are extremely difficult to carry out and which require for their development a constant retention of the doctrine of the phase rule and of reactions which take place in order that the conclusions which he draws may be sound. Another interest that we have in the tungsten carbide system comes from the fact that tungsten in vacuum tubes is frequently carburized. The work which Mr. Sykes has described for you today has a very interesting and important bearing on that field.

Dr. Grossmann has pointed out fields of future work. Not only is the iron-carbon-tungsten system of great interest, and one which would logically follow the work on the binary system, but also the cobalt-tungsten-carbon system is of great interest. Yesterday afternoon we had a paper which dealt specifically with the action during cementing tungsten carbide and that investigation shows how closely that cementing action ties up with equilibrium between cobalt and tungsten carbide at elevated temperature, and I do not believe that it is a very far cry from cemented tungsten carbide in which cobalt is used to cement the tungsten carbide.

[2]Research metallurgist, General Electric Co., Schenectady, N. Y.

CEMENTED TUNGSTEN CARBIDE

By Gregory J. Comstock

Abstract

Two years of general application of cemented tungsten carbide have conclusively demonstrated its commercial value. The history of its introduction has been somewhat similar to that of high speed steel. Cemented tungsten carbide is a product which was developed in the electric lamp industry and consists of hard particles of tungsten carbide cemented in an alloy matrix. It is produced by purifying tungsten ore; reducing tungsten oxide to the metal; carburizing the tungsten; grinding the carbide; milling it with cobalt; compacting the mixed powders; shaping; and first and final sintering. Heating operations are conducted under hydrogen.

The physical properties of cemented tungsten carbide are, high hardness, high heat conductivity, and low coefficient of expansion. Its strength varies according to the grade (percentage of binder and particle size) from 180,000 to 350,000 pounds per square inch. Its compressive strength is above 500,000 pounds per square inch.

Immediate improvements will be the introduction of a number of grades which will be produced by methods which will permit regulation of grain size in the finished product and possibly the introduction of metals other than cobalt in the alloy binder.

TWO years of general and increasing application of cemented tungsten carbide to the industry of this country have conclusively demonstrated but have not as yet entirely defined and evaluated the commercial value of this unusual product. Thus far the history of its application has been similar to that of high speed steel, which was one of high anticipation; alternating discouragement and encouragement and finally of successful and wide-spread utilization. High speed steel at its inception however, had one decided advantage over this new material in that it was a product

A paper presented before the Twelfth Annual Convention of the society, held in the Hotel Stevens, Chicago, September 22 to 26, 1930. The author is a member of the society and is director of research for the Firth-Sterling Steel Co., McKeesport, Pa.

of a well recognized method of manufacture. Since time immemorial we have produced metal objects by the melting process—at first crudely, later skillfully, and finally intelligently. The unavoidable mystery and misunderstanding which has shrouded the manufacture and composition of cemented tungsten carbide has undoubtedly militated against its successful use in a great many cases. A thorough understanding of the ultimate composition and character of a new and revolutionary material is most certainly essential for its intelligent application.

Much has been said and written concerning the physical characteristics and unusual hardness of this alloy. Brief discussions of the methods used in producing it have been published from time to time by those who are engaged in its manufacture. Actual trial of the material itself has familiarized many with its possibilities when applied to individual problems. It is believed, however, that there yet remains the necessity for discussing and explaining the fundamental character of the material in sufficient detail to permit a general understanding of its unusual composition.

Many years ago Moissan when experimenting with his electric arc furnace produced an extremely hard substance by the fusion of carbon and tungsten oxides at high temperatures. Chemists were fortunately able to identify this compound almost immediately as being a carbide of tungsten. Comparatively recent investigations have more or less positively demonstrated that a portion at least of the cutting capacity of high speed steels is attributable to the presence of a multitude of well dispersed microscopic and sub-microscopic particles of the complicated carbides of tungsten, iron and chromium which are held or cemented in place by an austenitic, martensitic or austenito-martensitic matrix from which they have been precipitated as a result of the high temperature heat treatment approaching fusion which is commonly applied to this alloy.

Viewed in its simplest form cemented tungsten carbide consists of minute irregular particles of tungsten carbide, which are believed to be harder and more stable than the carbides present in high speed steel, cemented in a thin metal matrix. The tungsten carbide particles constitute roughly from 85 to 97 per cent of the metal aggregate. The carbides present in high speed steel cannot exceed a third of the amount present in the carbide alloy according to the same rough generalization. Regarded in this light it will be seen that the carbide alloy may be considered as the cutting

essence of high speed steel. It is perhaps reasonable to wonder why its fabrication was not attempted several years ago.

History of the Material

The original conception of a hard metal alloy composed largely of tungsten carbide was evolved in the electric lamp industry where the necessity existed for wire dies capable of withstanding the hard usage imposed by the manufacture of metallic tungsten filaments. The highly refractory nature of tungsten which precludes its satisfactory casting and working according to the methods which have been commonly applied to metals of lower melting characteristics, familiarized the pioneers of the lamp industry with a type of metallurgy which was peculiarly their own. The processes of this industry are largely founded upon the physical laws which govern the fusion of finely pulverized metals and the metallurgy envolved may quite fittingly be called "the metallurgy of powders." When extremely hard dies were required for the manufacture of filaments therefore, the powder metallurgist was able to make quite satisfactory ones by the methods with which he was most familiar, i.e., the fusion at high temperatures of tungsten, carbon and cobalt or iron. From this beginning has been evolved cemented tungsten carbide which is today practically the only product of a metallurgy which has, until recently, been confined to the manufacture of one particular product only but which now gives every indication of taking an important place in our manufacturing scheme. Emphasis should possibly be placed on the fact that powder metallurgy is at present in reality the metallurgy of powdered tungsten, or more recently, the metallurgy of powdered tungsten carbide. Its laws and processes are distinctly in the formative stage. Scientific interest and activity have been quickened however, by the commercial possibilities presented by the alloys which are made possible by these new processess and intensive investigations and study have now been going on for some time. It is safe to prophesy that the present alloy we are discussing is but the forerunner of many other valuable products.

Methods of Production

The methods which are employed in the manufacture of cemented tungsten carbide alloys which are now available for use

as tool and die materials may be described in sufficient detail to give an adequate idea of the ultimate composition and character of the material itself.

The process includes the preparation of extremely fine tungsten powders of high purity; the carburization of tungsten powders to produce tungsten carbide; the mechanical comminution of tungsten carbide with powdered cobalt; the compacting or pressing of these mixed powders to billet form; the first or pre-sintering heat treatment; shaping; and final sintering.

Either tungstic acid WO_3 or ammonium para tungstate $(NH_4)_{10}W_{12}O_{41}.11H_2O$ is prepared from carefully selected ores by the chemical processes which are commonly employed in the preparation of these compounds. When Sheelite is the ore used the preliminary treatment consists of decomposition with a commercial acid. During this treatment the calcium goes into solution and crude tungstic acid remains as a sludge. The reaction may be represented by the equation:

$$CaWO_4 + 2HCl = WO_3 + CaCl_2 + H_2O$$

The finely ground ore has been previously mixed with a small quantity of manganese dioxide which completely oxidizes any iron present during this reaction. The crude tungstic acid thus produced is first purified by reprecipitation, being dissolved by the addition of ammonium hydroxide and after filtering, reprecipitated by the addition of hydrochloric acid. An exact description of this process may be found by those interested in a treatise on tungsten by Colin J. Smithells published by Van Nostrand. The purified salts of tungsten are next reduced to the metal in tube furnaces which contain an atmosphere of hydrogen at a temperature which is carefully regulated to produce a minimum grain size. The result of this reduction is a fine metallic powder, the grain size of which may vary between two and six μ and the purity of which is rigidly kept above 99.5 per cent.

The metallic tungsten is then carburized by any one of several well-known methods which consists either of passing the powder through a furnace containing a carbonaceous gas or by heating it under hydrogen when intimately mixed with pure and finely divided carbon. The result of the carburizing process is a finely divided powder consisting of tungsten carbide (WC) the

hardness of each particle of which has been determined as being between that of the sapphire and the diamond.

This powder is then milled in a stainless steel ball mill with powdered cobalt, which being soft, coats each particle of tungsten

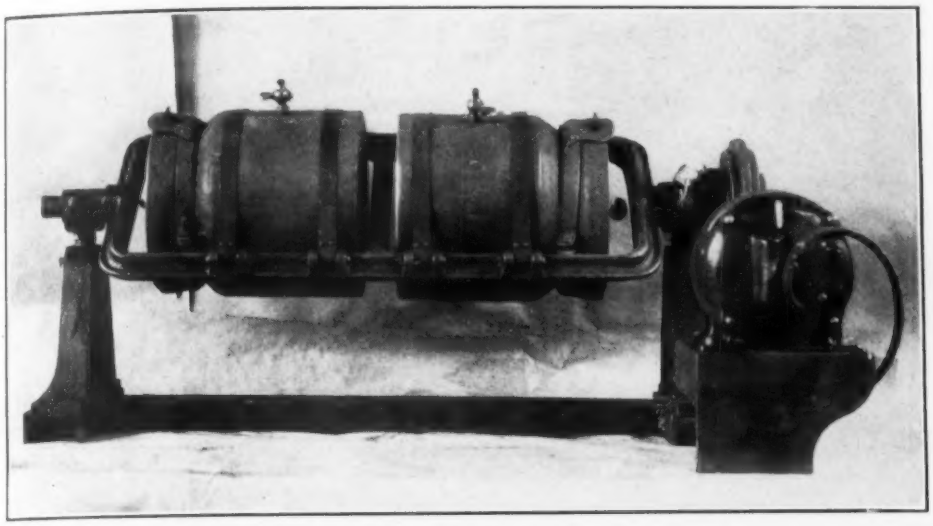


Fig. 1—Stainless Steel Barrels Used for Milling and Grinding Carbide and Cobalt Powders.

carbide more or less thoroughly with a thin pellicle of that metal. The milled metals are then ready for compacting.

This operation is performed in a steel die under hydraulic pressure. The powders are pressed into billets of suitable size and shape for the manufacture of tips for tools, dies for drawing wire, etc., etc.

These billets are next heated in hydrogen to a temperature which gives them sufficient cohesion to prevent breakage during shaping to the desired conformation. This heating operation is designated as the first or pre-sintering treatment. In the presintered state the material resembles graphite both in appearance and workability and may be shaped with a file or even turned in a lathe without fear of spalling or breaking. The increase in strength which results from this heating to several hundred degrees below the melting point of cobalt is attributable to the fact that thin impinging cobalt oxide films are reduced during the treatment and thus lightly cement the powdered metals together.

When shaped the pieces are final sintered at a high, white heat, again in an atmosphere of hydrogen. During this treatment cementation takes place, a marked change in size is noted and the

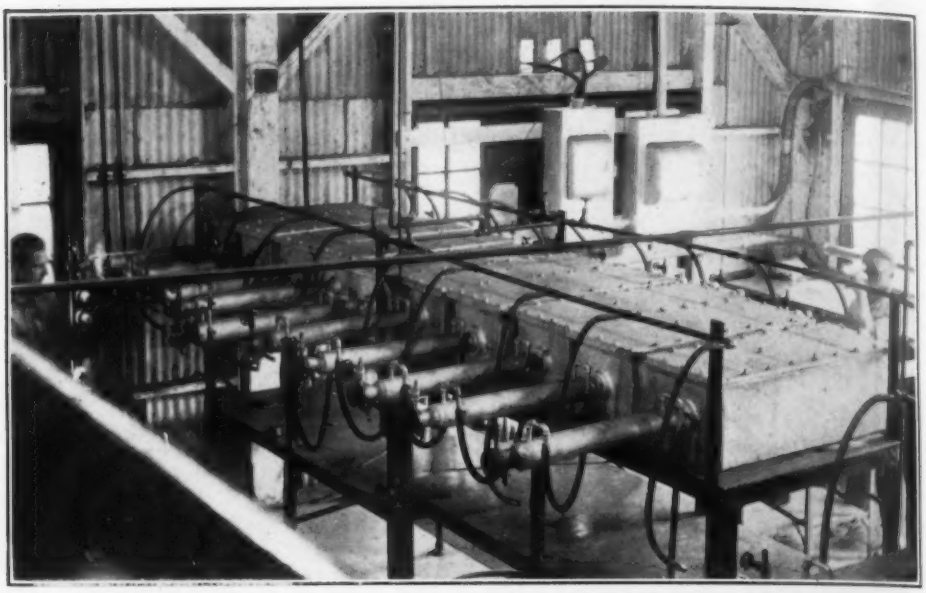


Fig. 2—A Battery of Molybdenum Wound Hydrogen Reduction Furnaces Used for Sintering Cemented Tungsten Carbide.

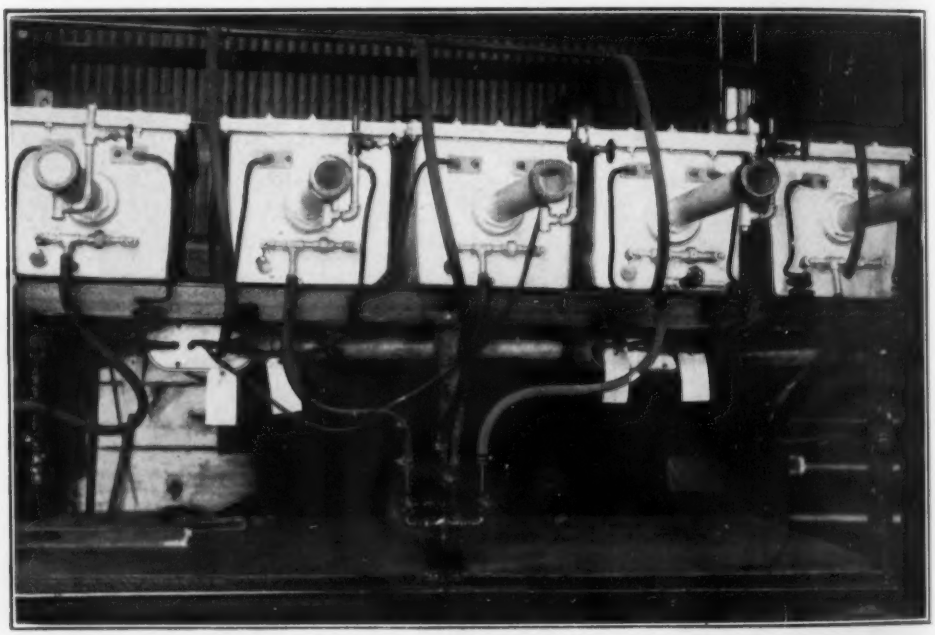


Fig. 3—Front Elevation of Hydrogen Reduction Furnaces Used for Sintering Cemented Tungsten Carbide.

piece which has previously consisted of a high porous mass of fine granular particles shrinks to a dense material which is without voids that can be detected at a magnification of 100 diameters. Except

for final grinding and perhaps lapping where a fine finish and sharp edges are desired the product is now ready for use.

Viewed in the light of our present understanding which is based on the results of considerable research we believe that the cementing action and marked shrinkage is due, in part at least, to the fact that cobalt dissolves some small quantity of the carbide during this operation and that the bonding action results from this

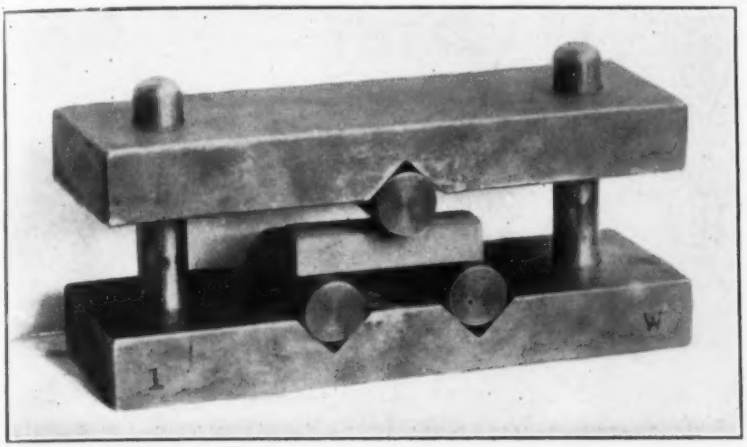


Fig. 4—Method of Testing Cemented Tungsten Carbide for Resistance to Transverse Rupture.

phenomena. Dr. Hoyt's recent investigations have quite positively indicated that a certain amount of tungsten carbide is reprecipitated on cooling and that the metallic cement consists of a eutectic alloy of carbon, tungsten and cobalt.

When fractured the finished material which has been prepared in this way resembles to the unaided eye fine grained high speed steel. When suitably polished and etched and viewed under a microscope at high magnification it will be seen to consist of small irregular particles of tungsten carbide which are closely adjacent to one another and are thoroughly bonded together with the alloy cement.

Properties of Cemented Tungsten Carbide

There is at present no satisfactory method of accurately determining the hardness of cemented tungsten carbide. As has been pointed out its abrasive hardness is the hardness of tungsten carbide, which has been calculated by German investigators to be 9.8 on Moh's scale of mineral hardness. The use of the Rockwell Hardness Tester as a means for determining the hardness of

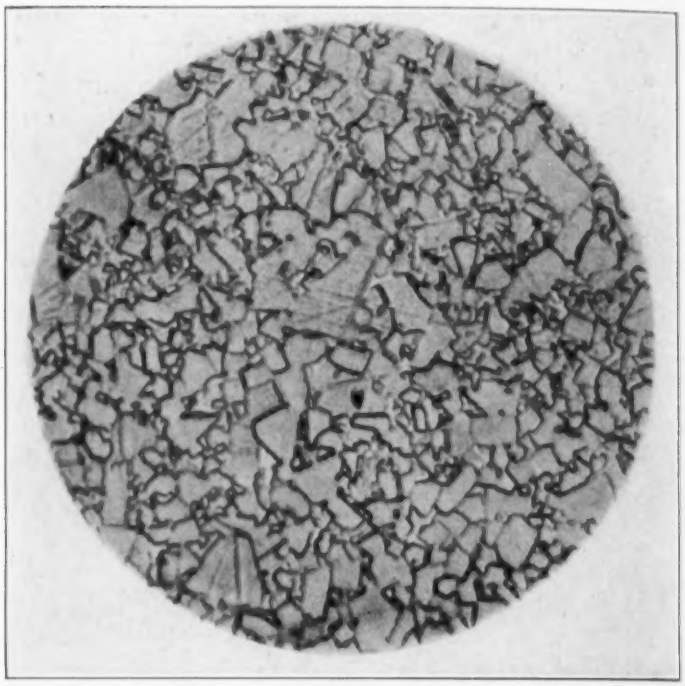


Fig. 5—Photomicrograph of Low Cobalt Cemented Tungsten Carbide Etched with Alkaline Ferricyanide Followed by Caustic Electrolytic Etch. L. B. Sykes in Author's Laboratory. × 1500.

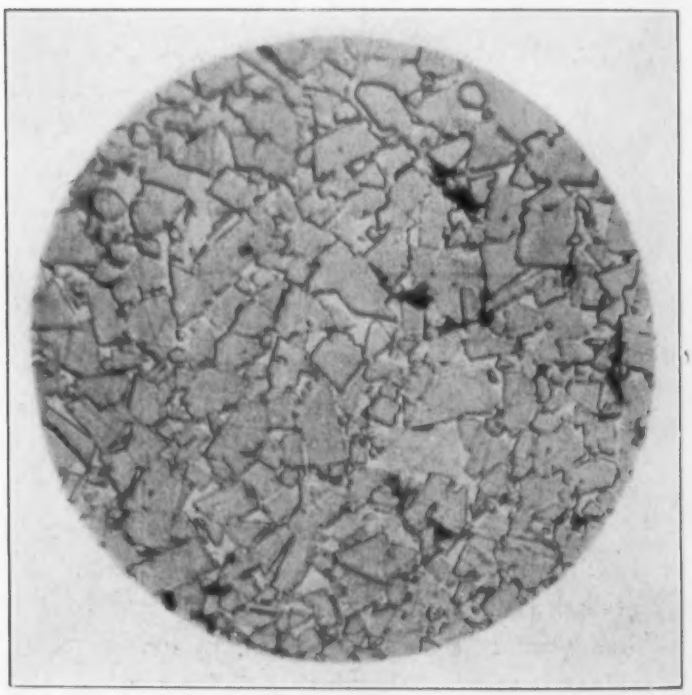


Fig. 6—Photomicrograph of Uniform Particles of Cemented Tungsten Carbide Etched with Alkaline Ferricyanide Followed by Caustic Electrolytic Etch. L. B. Sykes in Author's Laboratory. × 1500.

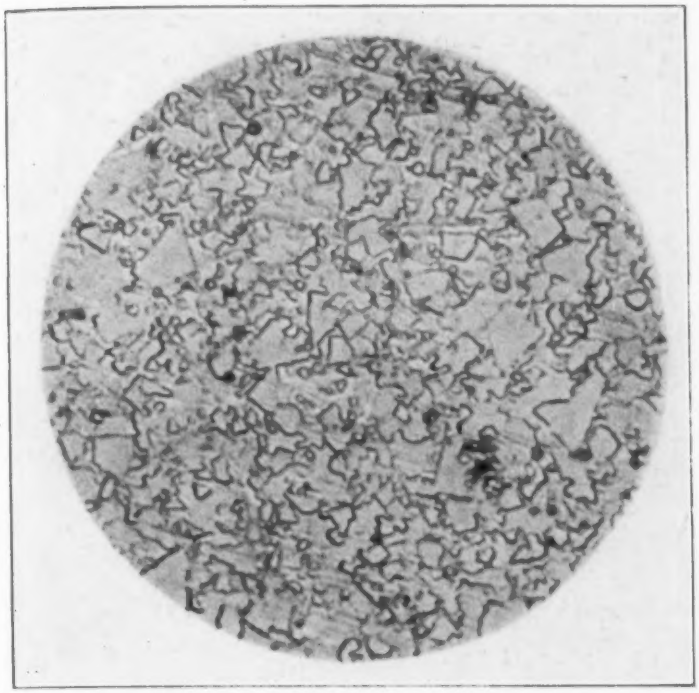


Fig. 7—Photomicrograph of 6.5 Per Cent Cobalt Cemented Tungsten Carbide Etched with Alkaline Ferricyanide Followed by Caustic Electrolytic Etch. L. B. Sykes in Author's Laboratory. × 1500.

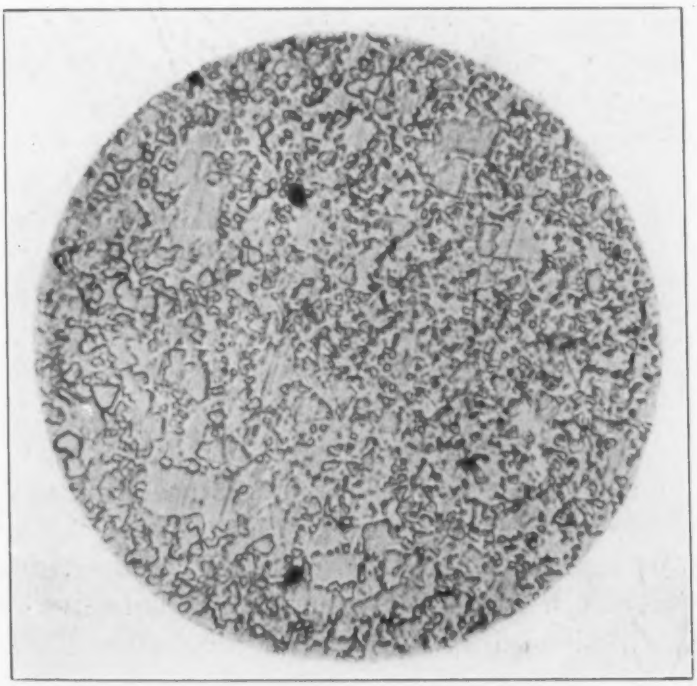


Fig. 8—Photomicrograph of Iron Cobalt Cemented Tungsten Carbide Etched with Alkaline Ferricyanide Followed by Caustic Electrolytic Etch. L. B. Sykes in Author's Laboratory. × 1500.

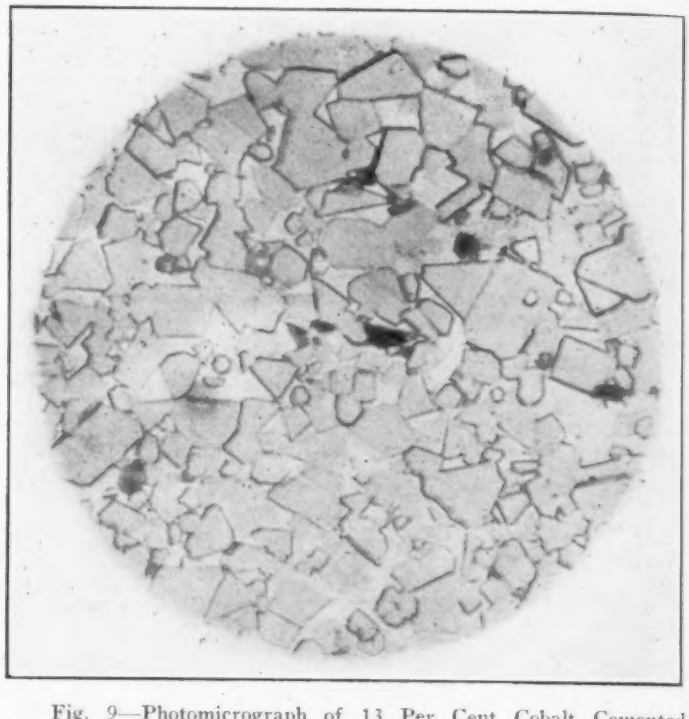


Fig. 9—Photomicrograph of 13 Per Cent Cobalt Cemented Tungsten Carbide Etched with Alkaline Ferricyanide Followed by a Caustic Electrolytic Etch. L. B. Sykes in Author's Laboratory. × 1500.

cemented tungsten carbide has been suggested and has met with some general application. The resistance to indentation of the cemented carbide does not give the true hardness of the material but is rather misleading as it measures in part, the strength of the bond, together with a hardness of the cement and the carbide itself. In lieu of better methods of testing, however, the Rockwell hardness tester can be used.

The heat conductivity of cemented tungsten carbide is approximately 0.21 in cm. gm. seconds. The coefficient of expansion is 4.4×10^{-6} the electrical conductivity is roughly 5 ohms per millimeter squared. The magnetic permeability has been determined as being 2. Cemented tungsten carbide may be expected to decompose at approximately 1600 degrees Cent. When subjected to cross-breaking stresses test pieces give a resistance of from 180,000 to 350,000 pounds per square inch depending upon the grade of the material which is being tested. The compressive strength of the material has been determined as being more than 500,000 pounds per square inch.

Grades of Cemented Tungsten Carbide

There are at present several grades of cemented tungsten carbide available for commercial use. These grades vary materially in hardness as measured by indentation tests and in toughness as shown by transverse breaking tests. These variations in physical properties were at first produced only by changes in composition, but are more recently being brought about by varying the size of the carbide particle. The application of a variety of carbide alloys to the commercial problems presented represents the next step in the development of this new type of metal aggregate.

Increasing the amount of bonding cement results in a toughening of this material but necessarily decreases the number of hard carbide particles present in a unit area of cutting edge or abrasion resisting surface. It may therefore be regarded as being one of the poorest ways of strengthening the product. More encouraging results have been obtained, however, by producing material, the carbide particles of which have been sized according to definite proportions. This method was suggested by the similar problem presented to those interested in producing stronger concrete and the development of a modulated stone size to obtain increased material strength. At least one grade of commercial cemented carbide is at present being manufactured according to this method. Investigations are in progress as to the possibility of further increasing the resistance of the material to transverse rupture by particle size regulation.

The introduction of small amounts of metals other than cobalt and the production of alloy binders presents a further possibility of the development of special grades of this material. Many such alloys have been studied and discarded as being undesirable, chiefly, it is believed, because the fundamental laws governing the tertiary alloys of tungsten, cobalt, and carbon have yet to be fully investigated. W. P. Sykes' excellent work on the alloys of tungsten and carbon undoubtedly aid this phase of carbide development most materially.

One interesting and as yet unexplained fact which has been noticed in the development of this material is that the tungsten which is produced from certain ores communicates most unusual and desirable properties of toughness combined with hardness to the finished product. The most carefully conducted analyses of the

various tungsten powders fails to reveal differences in composition which are sufficient to explain the results which are obtained.

Later investigations will doubtless throw light on this subject. At the present time, however, at least one grade of commercial cemented tungsten carbide is being produced from selected ore and although its composition is a usual one, its physical characteristics and performance results are sufficiently unusual to warrant its classification as a distinct grade of carbide alloy.

Uses of Cemented Tungsten Carbide

The performance of cemented tungsten carbide tools and dies is in many cases surprising even in view of the unusual character



Fig. 10—Photograph Showing the Use of a Tungsten Carbide Tipped Tool in Turning Aluminum Bronze at the Rate of 425 Feet Per Minute with a 0.024-Inch Feed and 3/16-Inch Cut, 50 Pieces Per Grind.

of the material itself. As has been indicated it is a reasonably strong and intensely hard alloy which is capable of being heated to comparatively high temperatures without material change, either in hardness or dimension. As such, it is undoubtedly adaptable to a great many commercial applications.

When correctly applied as a tip to a tool steel shank and

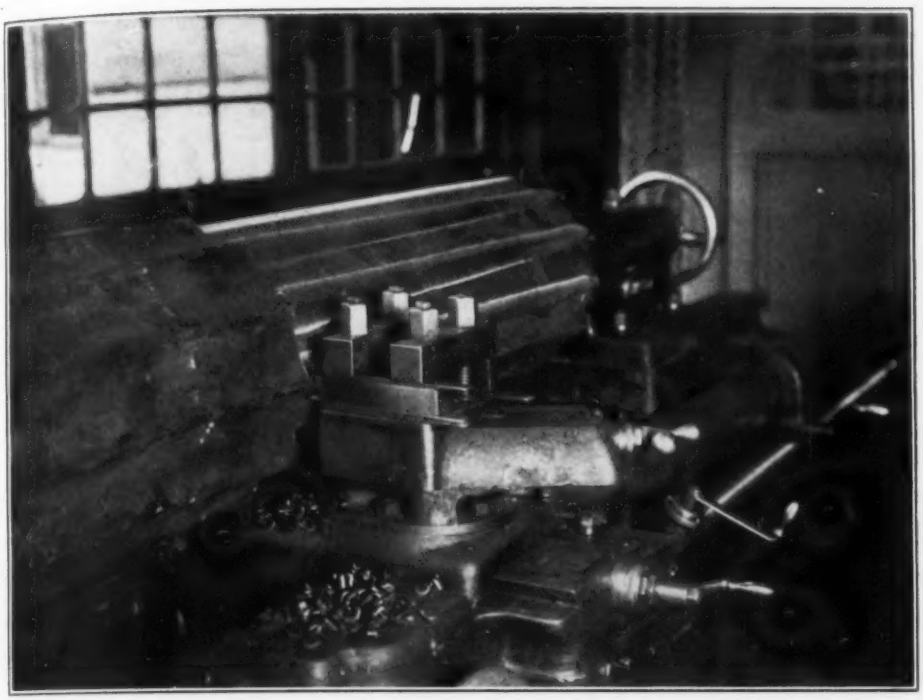


Fig. 11—Photograph Showing Turning a Fluted Steel Ingot With a Tungsten Carbide Tipped Lathe Tool, 50 Feet Per Minute, ⅛-Inch Cut, 1/22-Inch Feed, 105 Minutes Between Grinds.

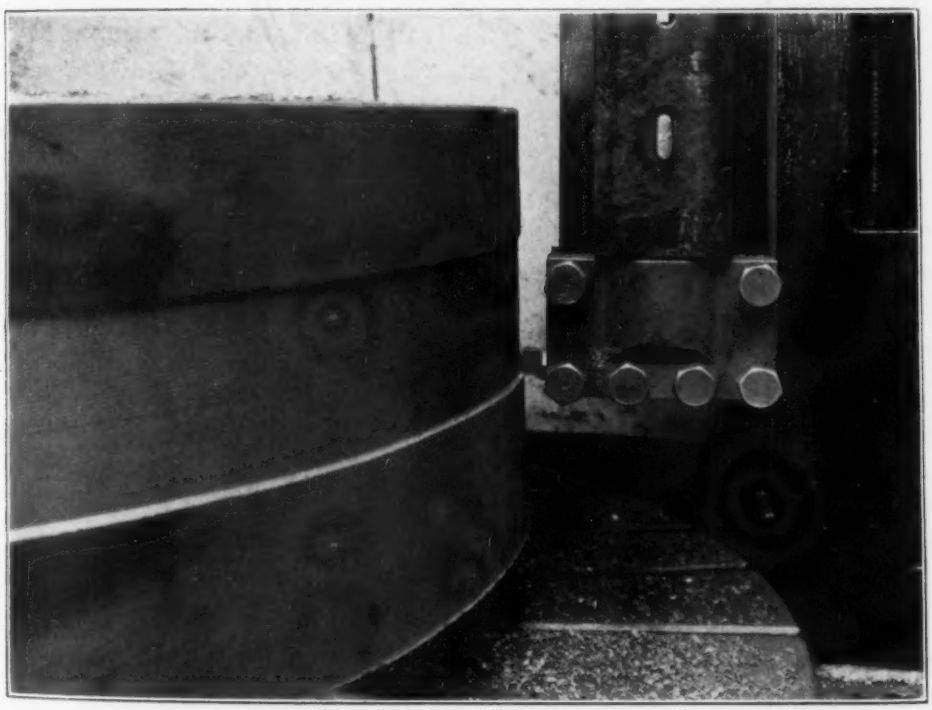


Fig. 12—Photograph Showing Turning a Large Cast Iron Fly Wheel with Tungsten Carbide Tipped Tool 1/11-Inch Feed, 175 Feet Per Minute, 1/2-Inch Cut.

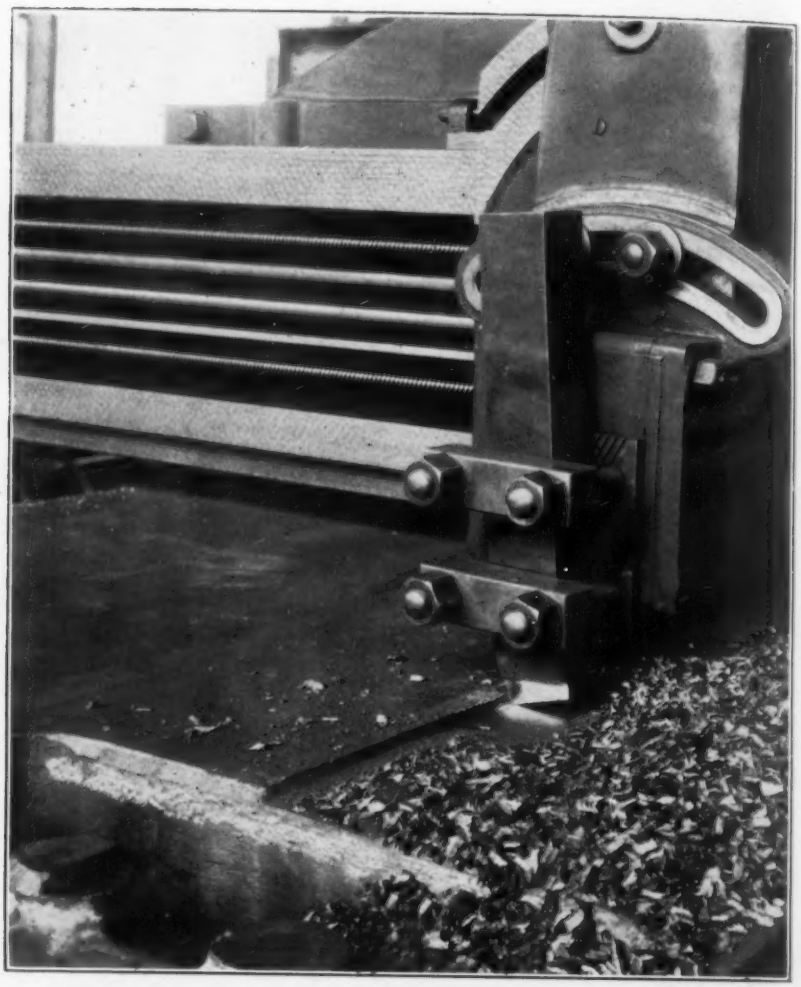


Fig. 13—Photograph Showing Planing a Semi-Steel Slab with Tungsten Carbide, 150 Feet Per Minute, 0.140-Inch Feed, $^1/_4$-Inch Cut.

ground to the most advantageous shape the carbide alloy has been found to possess a great capacity for cutting a wide variety of substances under speed conditions which have hitherto been regarded as being impossible.

Molded to suitable shape in the process of its manufacture and used as a die for drawing wire cemented tungsten carbide has been shown to possess attributes which are responsible for production results that are comparable to those of a diamond used under similar conditions.

In the application of this new product to cutting tools considerable experiment and trial by error has been found necessary, possibly because the true character of the alloy was not sufficiently

considered or understood. As the carbide is expensive it is applied as a tip to the cutting tool, a somewhat difficult matter owing to the differences in coefficient of expansion, heat conductivity and hardness that exists between the carbide and the tool steel shank to which it is bonded. With the increased cutting speeds which are made possible by the use of carbide, new tool shapes have been found necessary if the best results are to be obtained or in many cases actual failure is to be avoided. The expense involved in experimenting sufficiently to overcome these difficulties has been considerable and has proved discouraging to many possible users. This experimental period is passing and there are now many machine and cutting operations which have been definitely assigned to tungsten carbide tools. In these installations tungsten carbide tipped tools are cutting as consistently day in and day out as are any of the other commercial alloys which are now available for our use and are affording materially increased production.

As has been mentioned, sintered tungsten carbide alloys were originally designed for difficult wire drawing operations. The application of the present commercial carbide alloys in this field is now rapidly being accomplished and it is safe to prophesy that in a very short time, indeed, its use will be general for certain types of wire, rod and even tubing. The saving involved is largely that of the time which was previously required to change rapidly worn out nibs and dies. In at least one of the many new carbide die installations it is now the practice to weld one coil of wire to the next and in this way to make practically continuous an operation which was previously subjected to a series of more or less constant interruptions. Users of these dies are most enthusiastic concerning them once they have worked out the necessary changes in die conformation which are required by the new material.

Innumerable concrete examples of these and other successful applications of cemented tungsten carbide could be stated but it is believed that they are rendered unnecessary by the fact that almost every one is obtaining first hand information on this subject.

Future of Carbide Alloys

In attempting to forecast the future of this material it may be best to regard it as the first product of a new metallurgy. In this age we have all of us become increasingly familiar with new and revolutionary improvements in practically all of the machinery of

our civilization. In the past, almost without exception, such betterment has come about by intelligently studying the older methods of our production and changing them in the light of the new understanding which has resulted from such research. In this way, for example, better and more efficient alloys have previously been produced. There must, however, of necessity be an end to the refinement which can be applied to any existing process. Even though we are most certainly far from accomplishing all of the improvements which can be made in our present process we should welcome any basically new and hitherto unexplored method of production. The fact, that the first product of this new metallurgy has proved to be so strikingly unusual both in its composition and results, should encourage us to believe that it will be followed by improvements which will be of still greater commercial interest. Two years of application have demonstrated the fact that there is an important place for cemented tungsten carbide in our industry even though those years have been largely spent in experimental application and in learning how the material should be used. During these two years much has been learned about the manufacture of the material itself and a sound beginning has been made in the study of the fundamental laws which govern the metallurgy of powders.

The immediate future of cemented tungsten carbide is most certainly the development of different grades of the material to meet the individual requirements of its diversified application. The future of sintered products in general and the effects they may be expected to have upon our manufacturing operations is, indeed, most difficult to forecast.

DISCUSSION

S. L. Hoyt:[1] One thing that appeals to me is that this paper was included in the program on carburizing which is in itself a cementing operation, although not a complete one. The cementing action that Mr. Comstock has described is of course quite a different metallurgical operation, and I am in quite complete agreement with what he says. Mr. Comstock has also taken us a little further into some of the details of the manufacturing operations and he has given you a very good picture of that phase. Mr. Comstock has also called attention to the peculiarities of this branch of metallurgy and of the trials and viscissitudes that a new product must necessarily go through on being put to general use. However, I think Mr. Comstock will have to raise some more controversial features before I shall have any real discussion to make.

[1]Research metallurgist, General Electric Co., Schenectady, N. Y.

THE MICROCHARACTER

Its Application in the Study of the Hardness of Case-Hardened, Nitrided and Chrome Plated Surfaces

By Christopher H. Bierbaum

Abstract

The importance of having definite information concerning the physical properties of microscopic formations or crystals, of both metals and alloys, has long been realized; among these properties none seems to be of more importance and more difficult to determine than their hardness. Skillful microscopical examinations of both broken tensile and compression test pieces definitely show their weakest formations; while, on the other hand, no adequate knowledge of the hardness of these individual particles can be gained in this aggregate manner. The hardness of an individual microscopic particle can only be determined by an individual test of the same. Investigators, in their efforts heretofore, have been strikingly handicapped by the crudeness of the means available for such studies. The more recent accomplishments in the grinding of the hardest of all substances to a degree of refinement never heretofore attained places entirely new means at our disposal.

THE need for having more refined means for testing materials than those now generally available has long been felt by many investigators, especially so for testing crystals and areas of microscopical dimension and also for testing exceedingly thin sections of metals. An instrument was designed by the writer[1] that should meet the requirements for testing microscopic areas, and should also serve for testing the hardness of extremely small formations, more especially with a view of studying the bearing alloys.[2] This same instrument, with the introduction of a harder jewel, is now equally well adapted for studying the hardness of exceedingly thin sections.

[1] *Mechanical Engineering*, p. 71, January, 1919.

[2] Study of Bearing Metals, A. I. M. E., February, 1923.

A paper presented before the twelfth annual convention of the society held in the Hotel Stevens, Chicago, September 22 to 26, 1930. The author is a member of the society and is vice-president and consulting engineer, Lumen Bearing Co., Buffalo, N. Y.

The procedure, briefly, consists in moving a highly polished and lubricated surface of the material to be tested beneath a very accurately ground jewel point, under a definite pressure with micrometer feed. It is essential that this cutting point be elastically held and that the movement be so slow that no additional penetration is effected by stopping. After a cut or a continuous indentation is made, its width is measured at desired points and the hardness determined according to a convenient formula.

A very considerable amount of study has been devoted to the shape and proportions of a suitable cutting point, not only as to the best and most efficient for such service requirements, but also with a view of enabling the duplication of a point under exact specification. After a somewhat prolonged investigation as to shapes, proportions and angles for such a cutting point, the corner of a cube was finally adopted as being the most desirable, both for serviceablity and also for offering the possibility for exact duplication.

The jewel that was first considered fairly satisfactory was the artificial leuco sapphire, fused Al_2O_3, if for no other reason, it was the hardest jewel which, up to that time, any lapidarian had been able to grind, approaching the degree of accuracy required. The usefulness of the sapphire unfortunately was found to be rather limited, in that a very slight and almost imperceptible wear of the point renders the same unfit for corroborative results, it also proved rather short-lived for many of the harder metallic compounds. The sapphire does not cut manganese and the hardest metals, neither the hardest nitrided nor the hardest chromium plated surfaces. Since the areas tested with this instrument are so minute, the results seem neither comparable nor consistent with any other present method of hardness testing, in which an area of appreciable size is brought under consideration and where composite results from a number of different grains or crystal are brought into play. The one material on which comparisons could be made would be a theoretically homogeneous substance; this, however, may have a theoretical existence only.

In order that the width of cut shall in all cases be a direct function of its depth, and the square of the width of cut be directly proportionate to the cross sectional area of such a cut; it is necessary, where the corner of a cube is selected, that the cutting point be exceedingly accurate, that the three facet be true plane surfaces, that the three lines of intersection of these three facet be straight

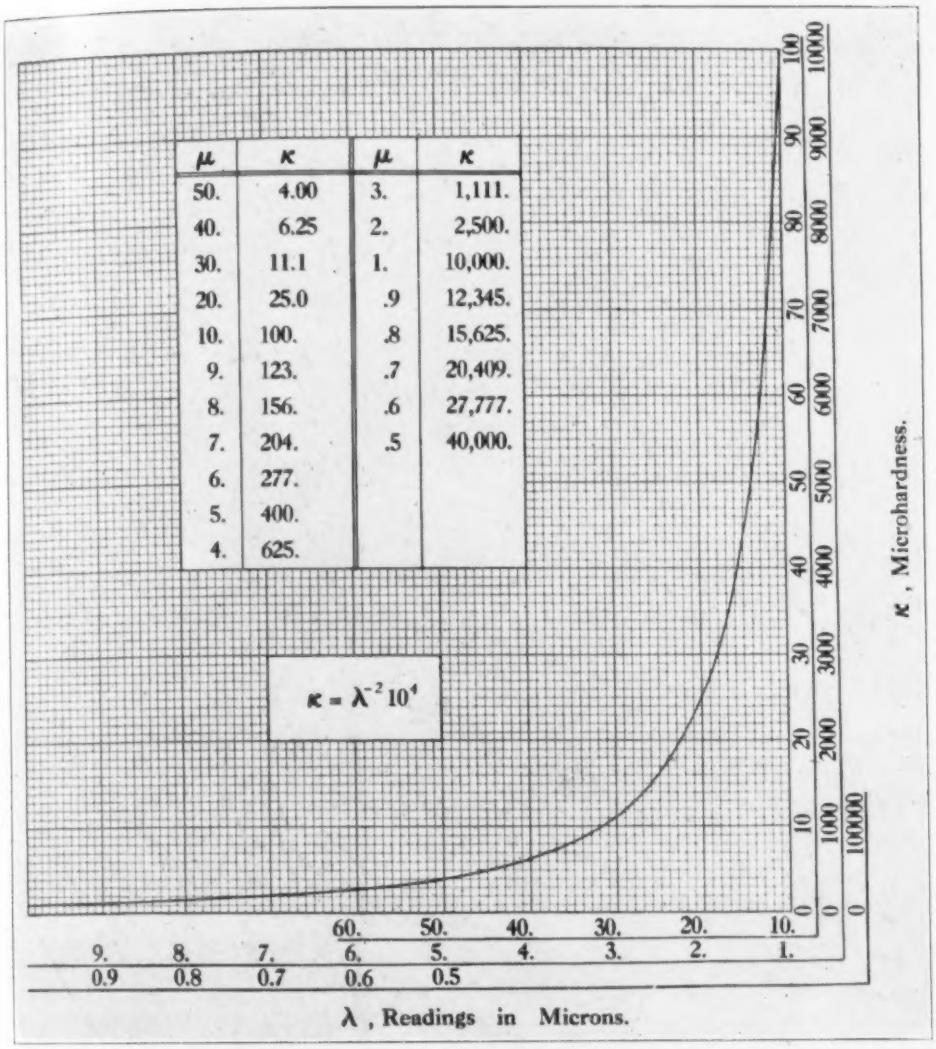


Fig. 1—Scale of Microhardness. This Curve is Plotted on Two Rectangular Co-ordinates. The Readings of the Widths of Microcuts are Given as the X-axis in Terms of Microns and the Corresponding Degree of Microhardness is Given on the Y-axis.

lines and that the point be without bluntness. The foregoing conditions are absolutely necessary in order that a rational scale of hardness may be established, one that shall apply equally well to all the degrees of hardness of the various metals and thereby give accurate means of comparison for hardness. In order to make the work of different investigators of general value, it is also necessary that their results and data should be directly comparable, but this can only be accomplished by having like instruments of the highest degree of perfection. After fifteen years of effort and experimentation,

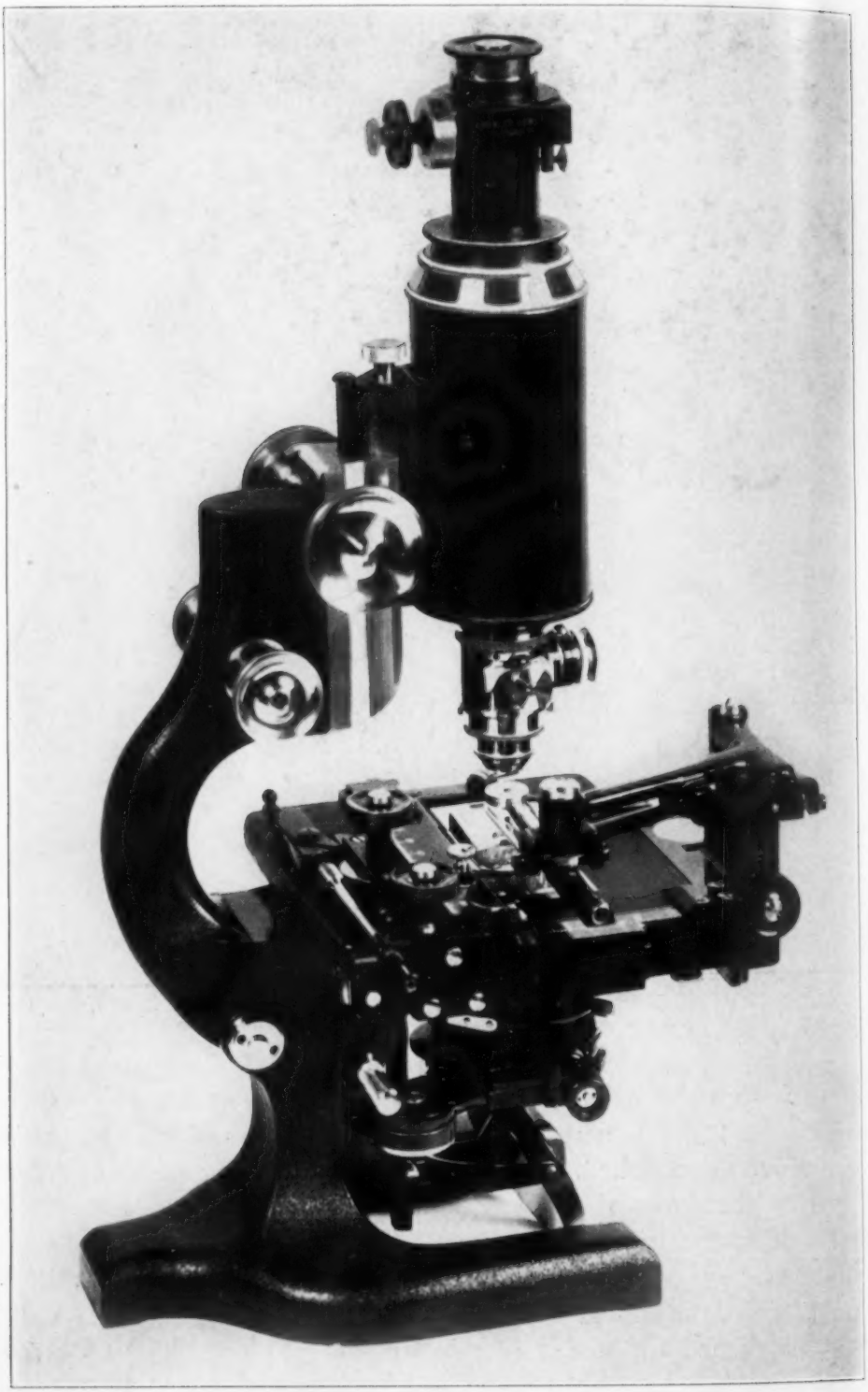


Fig. 2—Complete Assembly on Microscope.

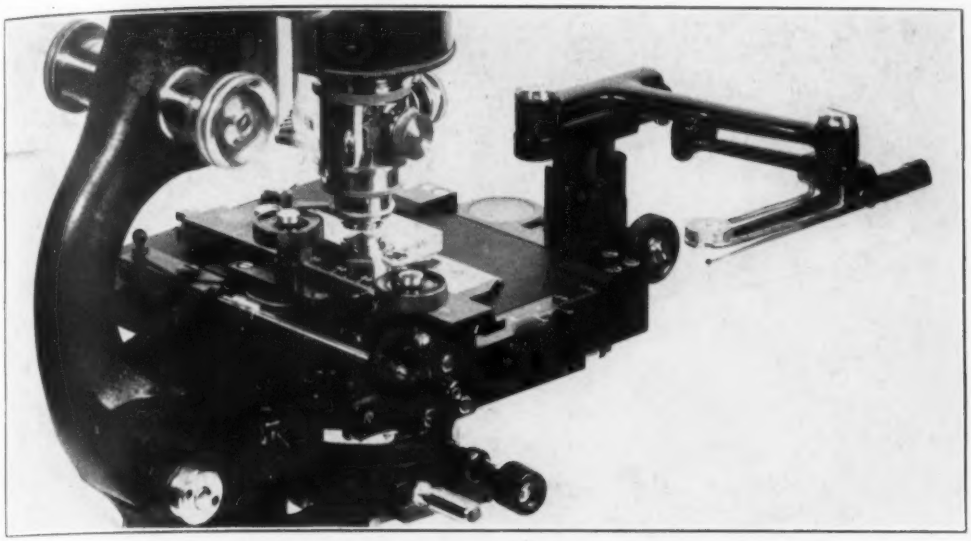


Fig. 3—Showing Jewel Bracket Swung Out of Position.

diamond points with the required degree of accuracy have now been ground.[3] These diamond points were found to be accurate when examined under a magnification of 2000 diameters. The facet were true plane surfaces, the three intersections formed by them were straight and smooth lines and each facet was a true right angle. These diamond cutting points are ground from very small, irregular stones, after the proper axis has been selected, they are ground with three facet; these three planes making a solid right angle and the point of intersection of the three planes constituting the cutting point, this was found to be without the slightest bluntness under the above magnification. The three facet are ground successively by turning the diamond about its axis through an angle of 120 degrees after each grinding and at the same time maintaining its axis at an angle of 35.246 degrees with the grinding surface. The diamond is then attached to the jewel spring in such a manner that if the solid

Table 1
Data for Chromium Tests

Specimen	Microhardness	Chromium Surfaces
1	725	Metallic Chromium
2	948	Chromium Plate 2/1000 in. thick
3	1900	Chromium Plate
4	4350	Chromium Plate
5	4400	Chromium Plate 1/1000 in. thick

[3]F. F. Gilmore, 112 Dartmouth St., Boston, Mass.

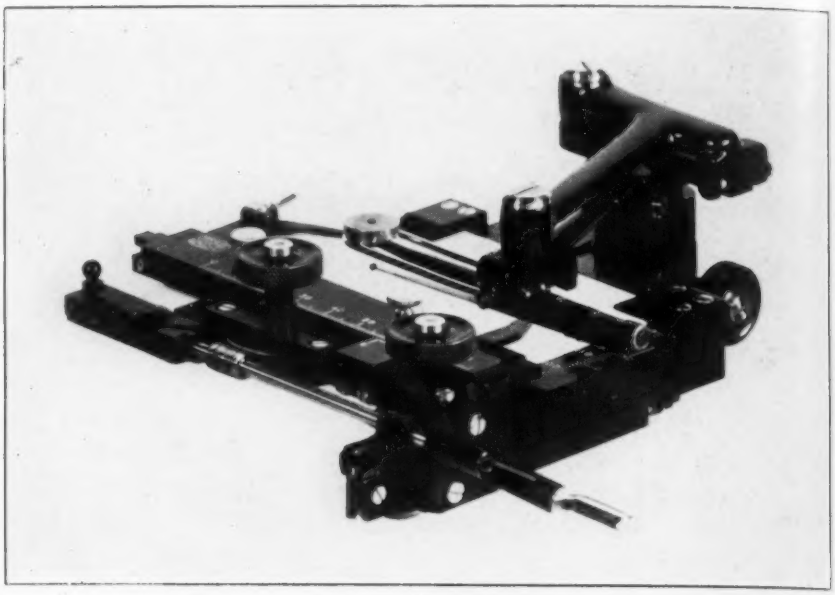


Fig. 4—Showing Microcharacter Separate from Microscope.

right angle or cutting point is considered as constituting one corner of an imaginary cube, the diagonal of this cube would then be normal to the test surface. One of the edges formed by the intersection of two facet forms the advance cutting edge and is in direct line with the cut. This advanced edge makes an angle of 35.25 degrees with the test surface, this constitutes the angle of incision. The depth of cut is always slightly less than four-tenths of its width. Care should be exercised when using the diamond to guard against accidental injury, since the diamond is a brittle substance.

The theory and procedure herein is quite different from that of other and earlier investigators, who have resorted to so-called "scratch methods". The present study, early in its inception, showed clearly that a clean, smooth cut made by a cutting edge held in an elastic support, gave the most uniform and accurate results. There should be no pulling or tearing to disturb even the smallest crystals from their natural location; that is, the vertical pressure should always be greater than the horizontal pull, a condition that obtains with the adopted cutting point, and the cut should be made with the least possible ragged and torn edges. Lubricating the surfaces of the test pieces adds very materially to the smoothness of the resulting cut. Of the large number of lubricants that have been tried so far, none have proven superior in any respect and none have

Number of Observation

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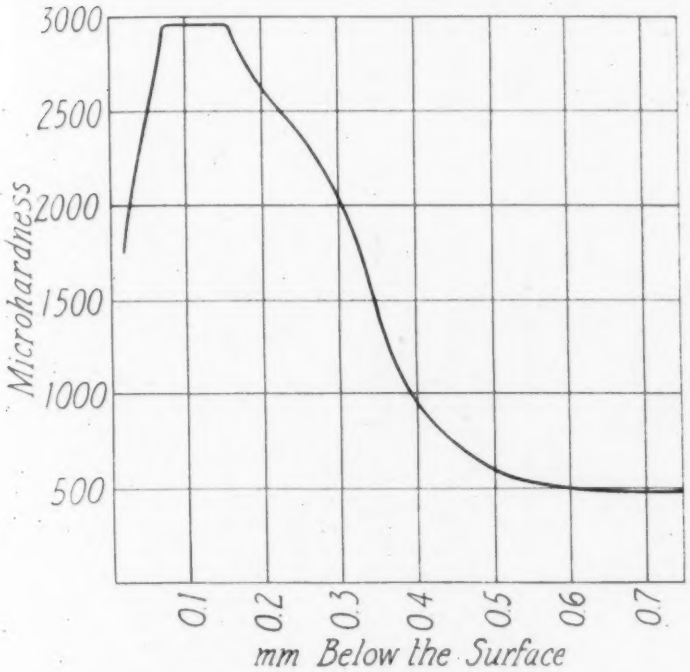


Fig. 5—Microcharacter of Nitrided Steel. Nitrided for 48 hours at 950 degrees Fahr. Dissociation 25 Per Cent. Flow 200 Liters per Hour.

Chemical Composition

C.	Mn.	Cr.	Mo.	Al.
.31	.63	1.12	.19	.93

Table II

Data for Nitrided Steel in Fig. 5.

Number of Observation	Distance from End of Specimen mm.	Depth below Surface of Case mm.	½ Width 9 gr. Microcut λ	Microhardness κ
1	.5	.0195	2.39	1800
2	1.0	.039	2.12	2250
3	2.0	.078	1.84	2950
4	3.0	.117	1.84	2950
5	4.0	.156	1.84	2950
6	5.0	.195	1.93	2650
7	6.0	.234	2.02	2450
8	7.0	.276	2.12	2250
9	8.0	.312	2.30	1950
10	9.0	.351	2.67	1450
11	10.0	.390	3.13	1050
12	11.0	.429	3.50	800
13	12.0	.468	3.77	700
14	13.0	.507	4.05	600
15	14.0	.546	4.32	550
16	15.0	.585	4.42	530
17	16.0	.624	4.51	505
18	17.0	.673	4.60	485
19	18.0	.702	4.60	485
20	19.0	.741	4.60	485
21	20.0	.780	4.60	485

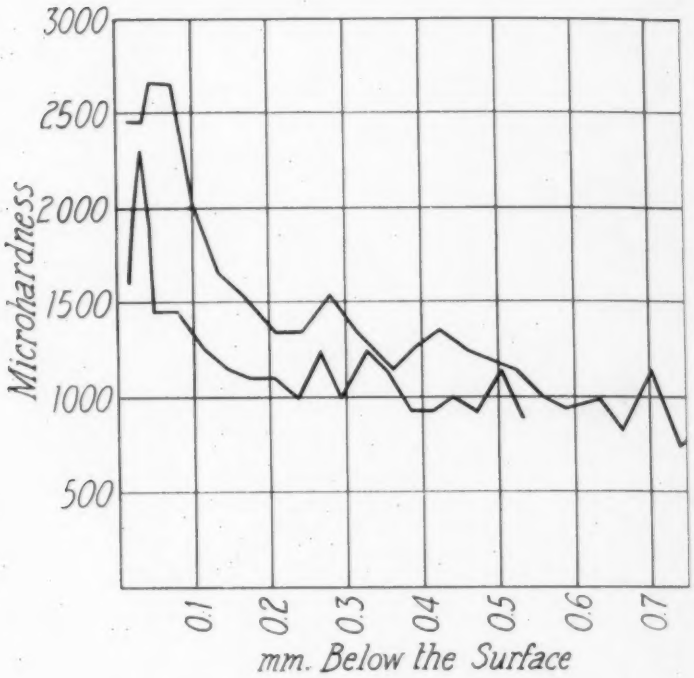


Fig. 6—Microhardness of Carburized Steels Upper Curve S. A. E. 4615 Steel Carburized 4 Hours at 1650 Degrees Fahr. Oil-Quenched from Pot. Reheated to 1450 Degrees Fahr. Oil-Quenched and Tempered at 300 Degrees Fahr.
Lower Curve S. A. E. 6115 Steel Carburized 4 Hours at 1650 Degrees Fahr. Water-Quenched from Pot. Reheated to 1450 Degrees Fahr. and Water Quenched. Tempered at 300 Degrees Fahr.

equalled, for smoothness of cut, the high grade animal oils sold as super-fine watch oil, the object being, of course, to maintain an oil film on the surfaces of the cutting point. The term "scratch hardness", therefore, does not apply even remotely to the present work and its use for that reason seems altogether ill advised. Since the degree and accuracy herein attained is equal to the limit of the modern microscope and in order to avoid confusion with that of other investigators, it seems altogether in keeping that the terms "microcut" and "microhardness" should be retained; also that the instrument has been properly named, the "mircrocharacter".[4]

For the most satisfactory results in this study, it is altogether necessary to have the highest grade of microscopical equipment. A rigid microscope with a fine adjustment, that is both delicate and responsive; the optical system should be the very finest obtainable, especially so in the study of the hardest metals and alloys; a 1.5

[4] μικρός, small and χαράσσειν, to engrave or cut.

Table III

Data for Upper Curve in Fig. 6.

Number of Observation	Distance from End of Specimen mm.	Depth below Surface of Case mm.	½ Width 9 gr. Microcut λ	Microhardness K
1	.5	.018	2.02	2450
2	.75	.026	2.02	2450
3	1.0	.035	2.02	2450
4	1.25	.044	1.93	2650
5	1.50	.053	1.93	2650
6	1.75	.061	1.93	2650
7	2.0	.072	1.93	2650
8	3.0	.106	2.30	1950
9	4.0	.141	2.48	1650
10	5.0	.176	2.58	1500
11	6.0	.211	2.76	1350
12	7.0	.246	2.76	1350
13	8.0	.281	2.58	1550
14	9.0	.316	2.76	1350
15	10.0	.353	2.94	1150
16	11.0	.388	2.85	1250
17	12.0	.424	2.76	1350
18	13.0	.459	2.85	1250
19	14.0	.494	2.94	1150
20	15.0	.529	2.94	1150
21	16.0	.565	3.13	1000
22	17.0	.590	3.22	950
23	18.0	.635	3.13	1000
24	19.0	.673	3.50	825
25	20.0	.706	2.94	1150
26	21.0	.741	3.68	750
27	22.0	.777	3.40	850

Table IV

Data for Lower Curve in Fig. 6.

Number of Observation	Distance from End of Specimen mm.	Depth below Surface of Case mm.	½ Width 9 gr. Microcut λ	Microhardness K
1	.5	.015	2.48	1600
2	.75	.022	2.30	1950
3	1.0	.030	2.10	2300
4	1.25	.037	2.20	2100
5	1.50	.044	2.30	1950
6	1.75	.052	2.67	1450
7	2.0	.059	2.67	1450
8	3.0	.089	2.67	1450
9	4.0	.118	2.85	1250
10	5.0	.148	2.94	1150
11	6.0	.178	3.04	1100
12	7.0	.207	3.04	1100
13	8.0	.237	3.13	1000
14	9.0	.266	2.85	1250
15	10.0	.296	3.13	1000
16	11.0	.326	2.85	1250
17	12.0	.355	2.94	1150
18	13.0	.385	3.31	925
19	14.0	.414	3.31	925
20	15.0	.444	3.13	1000
21	16.0	.474	3.31	925
22	17.0	.503	2.94	1150
23	18.0	.533	3.40	875

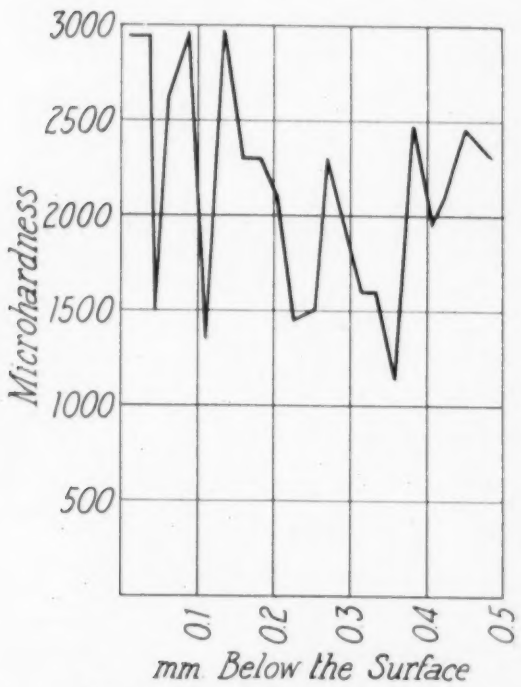


Fig. 7—Microhardness of S. A. E. 2512 Steel Carburized at 1650 Degrees Fahr. for 5 Hours Cooled in Pot, Reheated to 1450 Degrees Fahr. Quenched in Oil and Tempered at 350 Degrees Fahr. for 30 Minutes.

Table V
Data for Curve in Fig. 7.

Number of Observation	Distance from End of Specimen mm.	Depth below Surface of Case mm.	½ Width 9 gr. Microcut λ	Microhardness κ
1	.5	.011	1.84	2950
2	.75	.017	1.84	2950
3	1.0	.023	1.84	2950
4	1.25	.029	1.84	2950
5	1.50	.034	1.84	2950
6	1.75	.040	1.84	2950
7	2.0	.045	2.58	1500
8	3.0	.067	1.93	2650
9	4.0	.090	1.84	2950
10	5.0	.113	2.76	1350
11	6.0	.136	1.84	2950
12	7.0	.158	2.10	2300
13	8.0	.181	2.10	2300
14	9.0	.203	2.20	2100
15	10.0	.226	2.67	1450
16	11.0	.249	2.58	1500
17	12.0	.271	2.10	2300
18	13.0	.294	2.94	1150
19	14.0	.316	2.48	1600
20	15.0	.339	2.48	1600
21	16.0	.362	2.94	1150
22	17.0	.384	2.02	2450
23	18.0	.407	2.30	1950
24	19.0	.429	2.20	2100
25	20.0	.452	2.02	2450
26	21.0	.475	2.10	2300

mm apochromatic objective, together with a Filar screw micrometer eyepiece of only the best modern make should be used. This latter optical combination should be such that for the study of the harder materials a reasonably accurate measurement of one micron of width can be made. This is very important, since with the use of a microscope, having an inferior optical combination, nothing but unsatisfactory and unreliable results can be obtained. In studying the softer metals or the softer formation, the foregoing extreme requirements are not so essential, although ample magnifying power is desirable at all times. The optical combination should be calibrated in the usual manner by the use of a stage micrometer, thereby determining the actual value of the micrometer eyepiece scale divisions. The investigator should possess the skill and manual dexterity of an accomplished microscopist or that of an experienced physicist.

When making determinations of microhardness, the microscope should be accurately focused upon the test surface, wholly disregarding the microcut while focusing. The distance between the two outer lines, representing the extreme width of metal or material disturbance, caused by the cut, should be accurately measured. This constitutes the width of cut and is represented by the value of λ, in terms of microns. In some cases, it may be found that in the making of a cut, burrs or fins are formed at the edge of the cut, giving rise to confusion in the measuring of its width. In such cases it is desirable to either make a cut at right angles to the original and after careful analysis the proper reading can be determined; in other cases, merely brushing a test surface at right angles to the cut with the edge of a small piece of hard wood will often add materially to clarify conditions. It is a comparatively easy matter to determine relative values for different crystals or formations, a single microcut, made upon a polished surface, will give this data; since this is clearly shown by the varying width of cut. What is more difficult, however, is to obtain definite and exact readings that shall express and represent the true microhardness; for this, it is all important that the points between which the measurement is to be made should be determined accurately. In all cases painstaking care should be exercised at this crucial point.

The cross sectional area of a microcut is directly proportional to the square of its width; theoretically, therefore, the width of cut should vary in proportion to the square of the load upon the cutting point. Results of tests fully demonstrate that this condition

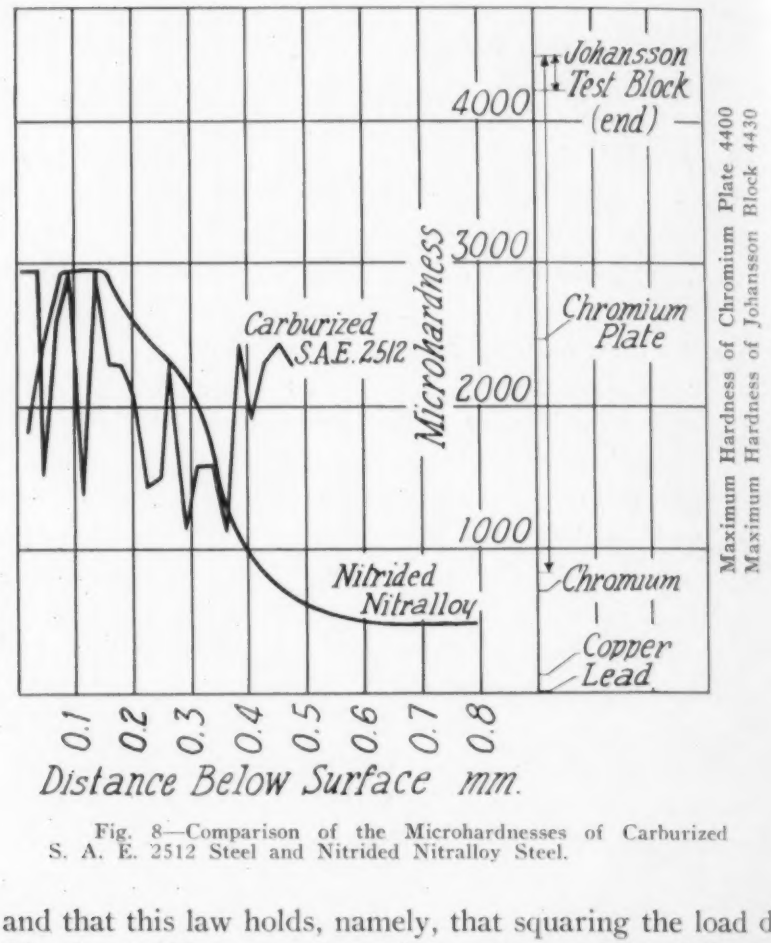


Fig. 8—Comparison of the Microhardnesses of Carburized S. A. E. 2512 Steel and Nitrided Nitralloy Steel.

exists and that this law holds, namely, that squaring the load doubles the width of cut. For general requirements, a weight of three grams seems to be very satisfactory and all calculations are made on that basis. For testing extremely hard surfaces, however, a nine gram weight offers distinct advantages, in that it produces a width of cut twice that of a three gram weight and the increased accuracy and convenience of reading the wider cut are well worth considering; after a nine gram cut is taken, its width is divided by two and referred to the following table of microhardness. An adjustment is provided by means of which the strength of the jewel spring can be changed from that required for a three-gram to that of a nine-gram weight. In every case after adjustment the jewel should be normal to the test surface and the jewel spring should not be brought in contact with the test surfaces. With these two weights, it is possible to test the hardness of all metals varying from that of pure lead to that of nitrided steel, chromium plate and the like.

Table VI
Data of the Microhardness of an 8 mm Johansson Test Block.

Cut 1 mm from End ½ Width 9 gr.			Cut 2 mm from End ½ Width 9 gr.		
Micrometer Eyepiece Readings	Microcut λ	Average κ	Micrometer Eyepiece Readings	Microcut λ	Average κ
1.6	1.47		1.6	1.47	
1.7	1.56		1.6	1.47	
1.6	1.47		1.6	1.47	
1.6	1.47		1.7	1.56	
1.6	1.47		1.8	1.65	
1.6	1.47		1.7	1.56	
1.7	1.56		1.8	1.65	
1.7	1.56		1.6	1.47	
1.7	1.56		1.6	1.47	
1.6	1.47	4430	1.7	1.56	4250

The scale of microhardness, as shown in Fig. 1, is a curve plotted on two rectangular coordinates; the readings of the widths of microcuts are given on the X-axis in terms of microns and the corresponding degree of microhardness are given on the Y-axis. The micron, a 0.001 of a mm, a unit commonly used in physics, seems to serve every convenient purpose as a unit of length and as such it is represented by the character μ. For the reason that practically all microscopical attachments are graduated in metric units, the metric system is herein retained for convenience. In developing a mathematical expression for microhardness, it is necessary to consider that the various cross sections of microcuts are symmetrical and have areas proportionate to the squares of their widths. These squares must, therefore, be taken into consideration; and also for the reason that the material which has the narrower microcut is the harder, therefore, the reciprocals of these squares should be considered. Now, if we let κ represent the microhardness and λ the reading for the different widths of microcuts in terms of microns, and then multiply the reciprocal of the square of the width of cut by ten raised to a convenient power, say 4, in order to avoid excessively long decimals, we have $\kappa = \lambda^{-2}10^4$. This formula has been plotted and is shown in Fig. 1. It is believed that this is the first rational scale of hardness, one that can express in definite units the relation of hardness between all the different materials.

Many of the alloys and purer metals show an appreciable variation of hardness depending upon the direction in which a polished surface is cut. This is a property of metals known as anisotropy, the property under which a single grain of pure metal or a single

grain of a solid solution alloy shows different degrees of hardness, depending upon the direction in which it is cut; this property is due entirely to the atomic orientation of the material and often adds in a varying degree to the complexity of this study. It is also one of the contributing factors why, upon a test surface, the microcut is not a mathematically straight line.

Fig. 2 shows a complete ensemble of a microscope with the necessary attachments for study. The setting shown is that necessary for making a microcut. After the same has been made, the jewel bracket is swung out of position, as shown in Fig. 3. The test surface is then cleaned of its lubricating oil with the use of xylol, the objective is then immersed in cedar oil and after focusing the width of cut is measured by the aid of the micrometer eyepiece. Fig. 4 shows the microcharacter separate from the microscope. In many cases, where two observers are working together, it is often convenient to have this instrument mounted on an improvised stand; the same having a stage substantially corresponding to that of the microscope stage in size and height above the working table.

The difference in hardness is very striking. Specimen No. 1 is pure metallic chromium, a sample originally supplied by the Goldschmidt Thermite Corporation. Nos. 2, 3, 4, and 5 are electrolytically chromium plated surfaces and the second column shows their respective degrees of hardness. Specimens 2 and 3 are apparently satisfactory products. Specimens Nos. 2 and 5 were plated in the same tank at the same time and on two steel strips cut from the same plate, with the instructions to the plater that he make one 1/1000 inch and the other 2/1000 inch thickness of plate, with results as shown. Specimens Nos. 4 and 5 are badly checked and have uneven surfaces. Can the hardest deposits be made without checks and unevenness?

Fig. 5 is a curve showing the results of microhardness tests of nitrided steel, the data as given in Table II. The hardness readings give the true value for each depth of case, since at the hardest point the penetration of the diamond is less than 1.5 microns (less than 1/17,000 inch) and in the softest core the depth of microcut does not exceed 3.7 microns (less than 1/17,000 inch), the cut having been made with a nine-gram weight and represents the deeper cut.

The striking result of the nitrided surface shown in Fig. 5 is its homogeneity; Fig. 7, in a carburized surface, shows crystals of the same maximum degree of hardness, but it also shows that a

distinct lack of uniformity of hardness exists. This is likewise true of the two surfaces of carburized steel shown in Fig. 6.

Microhardness tests were made on an 8 mm Johansson test block by making two microcuts across one end and in each case parallel to the end, one cut was 1 mm and the other was 2 mm from the end. The variations in the readings of the width of these microcuts almost approached the personal error of observation, showing that a very high degree of homogeneity exists in this steel, closely approaching that of the nitrided steel. The microhardness readings of this block are higher than the nitrided and carburized steels. The hardest chromium plate alone approaches or nearly equals the microhardness of the old imported Swedish Johansson blocks.

DISCUSSION

C. B. SADTLER[1]: I have listened to Mr. Bierbaum's paper with a great deal of interest. I am wondering how many there are here who, like myself, have hesitated tackling the microcharacter. I will say frankly that it took me about a year to convince myself that I could get into that sort of work and really produce results which would be of value in commercial work. I have only had the use of the microcharacter for a short time and there are a number of questions in my mind. I would like to ask one or two now. I will preface my remarks by the statement that my own feelings in regard to hardness testing are that we are on the verge of an era of more accurate testing. Without any disparagement of the old forms of testing, the scleroscope, Brinell, Rockwell and so on, I predict that within a short time we will be discussing smaller percentages of errors in individual measurements, or averages of a larger number in measurements, than has been heretofore possible. I consider good hardness testing, at least in the case of hardened steel, to be of the order of magnitude of a few tenths of a per cent precision. That kind of remark can only be made in the case of certain kinds of hardness results. It can be made in the case of Vickers testing. I am very much interested in knowing the precision that we may expect in microcharacter testing, in the case of the harder steels of the type of nitralloy and others, that is, in regard to measurements where we feel there is a high degree of uniformity of the material itself. I believe that Johansson block measurement referred to by the author could be taken as a high degree of uniformity. Unfortunately we cannot tell what the degree of uniformity is until we know what the precision of hardness testing is. But if we may assume a degree of uniformity I would like to have some idea of the value of the precision of hardness testing, using the microcharacter under the best conditions of operation.

A. B. KINZEL[2]: We have used this machine for some time as you know.

[1]Metallurgist, Barber-Colman Co., Rockford, Ill.

[2]Associated with Union Carbide and Carbon Research Laboratories, Long Island City, N. Y.

We have the earlier model with a sapphire. We agree with Mr. Bierbaum that the sapphire leaves much to be desired. It is necessary to periodically check it.

I should like to know whether the effect of using regular balsam oil as used on a microscope instead of watch oil is significant.

I should like to protest against the use of any formula to get a hardness number. As has been pointed out it is difficult to get a reading due to the plastic effect. If that reading is converted and another is taken under other conditions and converted no correlation necessarily exists. In our own work we give load and width of indentation and do not attempt to give a hardness number. The only exception has been in papers which we have published where it has been necessary to give more information so that the contents would be more easily understood.

D. LEWIS[3]: I would like to ask where such an instrument could be purchased or whether we have to make our own. Also whether the soft surface found on nitralloy is always present and whether it is more so in some cases than in others.

Written Discussion: By J. P. Walsted, instructor in physical metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

Mr. Bierbaum brings up the question of hardness testing. This has been in the past and will, no doubt, continue to be a fruitful source of argument. For careful scientific work the microcharacter machine leads the list. However, in fairness to other instruments, I would like to point out that the curves he obtains by the microcharacter and which he claims to be the true hardness depth curve has been known for some time. The same curve is obtained by the monotron in less time and just as accurately.

Author's Closure

There is no direct relation between microhardness and other methods of hardness testing; such as Brinell, Rockwell, scleroscope and Vickers, since the latter methods bring into play more than one micro-constituent in their tests. The object and purpose of microhardness testing is that of determining a physical property of the smallest possible microscopic constituent of a substance.

Its purpose is not that of replacing, nor is it that of coordinating present means by greater refinements.

In all of the established scales of hardness, it is universally customary to express the greater hardness by the higher numerical designation; accordingly, the microhardness of a substance should not be expressed in terms of microns denoting the width of the cut, since this leads to the inconsistency of designating the softer material by the larger hardness number. For this reason the reciprocal should always be used, and also for the reasons hereinbefore given, the hardness is inversely proportional to the square of the width of cut; therefore, the mathematical expression for microhardness should contain, as in all other expressions for hardness, a factor representing the reciprocal of the square of a lineal dimension of the indentation in such a test.

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[3]Research engineer, American Chain Co., Bridgeport, Conn.

The reason for lubricating test surfaces while making microcuts is that of securing as smooth a resulting cut as possible, lubricating distinctly aids in this respect. The superfine watch oils of purely animal origin are quite superior to the optical immersion or cedar oil.

So far all of our tests made on nitrided nitralloy steels distinctly show a softer outer surface. Between the different nitrided samples there is a wide range of maximum microhardness and the one hereinbefore shown is the hardest which as yet we have found. The characteristic feature in which a nitrided nitralloy surface differs from that of a carburized case is its high degree of homogeneity; it is not made up of relatively hard and soft crystals, as the carburized materials. The depth below the surface at which this maximum microhardness of a nitrided surface occurs seems to vary between 0.05 and 0.12 mm. The variation of microhardness of the chrome plate is of very considerable interest and is a subject which still seems to require some study.

The microcharacter carrying a certificate from the laboratories of the Lumen Bearing Company can be secured from the Spencer Lens Company, Buffalo, N. Y.

WEAR OXIDATION, A NEW COMPONENT OF WEAR

By Dr. Max Fink

Abstract

Wear of metals has generally been considered to consist of the mechanical removal of small particles and cold deformation and hardening under wear conditions. The author of this paper, after studying the effect of oxidation, has added what he considers another essential component of wear, this is called wear oxidation. Various wear tests are described including those conducted in inert atmospheres. It is shown that the wear effect of sliding friction can not be directly compared with the effect produced by rolling or with part rolling and part sliding friction.

IN HIS paper[1] G. M. Eaton says "comparatively little is known about the phenomenon surrounding the wear of metals." Hitherto two phenomena have been considered to be the essential characteristics of wear, (1) the mechanical removal of small particles and (2) the cold deformation and cold hardening of metal under wearing conditions.

The first characteristic, the removal of particles and therewith a loss of weight is generally known, especially in the case of sliding friction, as for instance the tire and brakes of railroad cars. The second characteristic, the cold hardening of metal during wear is especially mentioned in the paper of Meyer and Nehl.[2] By cold hardening, the metal increases in strength and loses in ductility. The cold hardening process takes place principally with rolling or sliding friction, for example the action of tire and rail in service.

The wear testing machines which have been built hitherto were constructed either with the point of view of duplicating the conditions of service or developing theories of the builders. In Germany

[1]G. M. Eaton, "Methods of Approximating Certain Physical Characteristics of Nitrided Steel Cases," Transactions, American Society for Steel Treating, January, 1929.

[2]Werkstoffausschussbericht 37, des Vereins Deutscher Eisenhüettenleute or *Stahl und Eisen*, 1924, p. 457-463.

From a thesis by M. Fink, submitted to the Technisch Hochschule Berlin-Charlottenburg, Germany, in partial fulfillment of the requirements for the degree of Dr. Ing. Completely published in the "Organ fuer die Fortschritte des Eisenbahnwesens," 1929, Heft 20. Okt. 15.

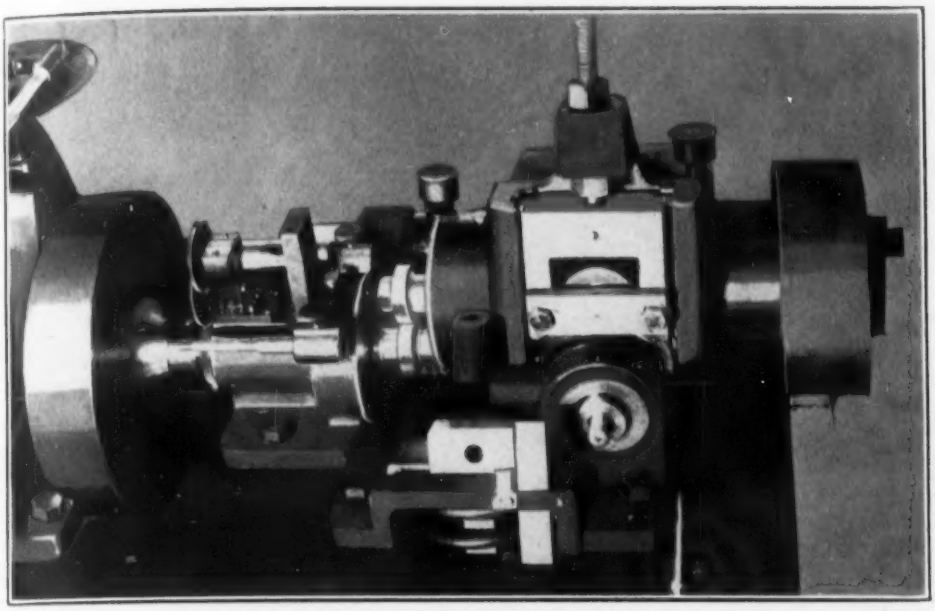


Fig. 1—Amsler Wear Testing Machine.

two kinds of wear testing machines are generally used. Wear testing machines with sliding friction (without emery and liquids) for instance the Spindel wear testing machine with a disk of mild steel, 350 mm. diameter, 1 mm. thick, rotating at 30 revolutions per minute, which is sliding and grinding in the test piece under a pressure of 5 kilograms. A measure for wear is the depth or the length of the segment ground away in a certain time or after a certain number of revolutions. Some wear testing machines use only rolling or rolling and sliding (or only sliding) friction without emery and liquids, that is for instance the Amsler wear testing machine. Here two disks of the metal to be tested are used, 40 millimeters diameter, 10 millimeters in length, rolling one upon another with a pressure of 50 or 100 kilograms, rotating at a rate of 250 revolutions per minute. The difference in weight before and after test, after a certain number of revolutions is the measure of wear. Fig. 1.

The results obtained with the two kinds of wear testing machines often do not check with each other and also do not simulate the results in practice. It is impossible as will be shown in this paper to compare the wear effect of sliding friction directly with the effect by wear with rolling or with part rolling and part sliding friction.

The wear debris or wear dust produced by the Spindel wear testing machine (sliding friction) is in an almost pure metallic condi-

tion while the debris of the Amsler machine with more rolling friction is oxidized and contains a great deal of FeO.[3]

It had been supposed hitherto that the particles dropping off the upper surface of the disks rest here and will be oxidized on account of their minuteness.[4] The oxidized dust has been regarded as a secondary phenomenon in the wearing process.

The presence of oxidized wear dust has been observed for instance in the case of the heavy application of brakes of a train, the particles will be torn off from brakes or tires with scintillation. The oxidation takes place however after the particles are torn off, the oxidation here is only a secondary appearance. In the English technical literature the oxidation during friction has been mentioned several times.[5] It was attributed in connection with corrosion phenomena to moisture or electrolytic effects or even to the stresses excited by friction. Tomlinson[6] who tried to reproduce the oxidation by friction with small balls on steel plates believes the cohesion to be the cause of small particles being lifted in a tangential direction out of the surface of the metal by the tangential forces during sliding friction and then the particles oxidize instantly.

On the Spindel machine with only sliding friction all of the dust or debris is metallic while on the Amsler wear testing machine with only rolling friction (and therewith a very small portion of tangential forces) the wear dust is completely oxidized. French[7] had observed oxide films too during the wear tests with bearing bronzes on an Amsler wear testing machine.

The researches of Meyer and Nehl on steel with a strength of 34 kilograms per square millimeter (48,350 pounds per square inch) and 61.7 kilograms per square millimeter (87,750 pounds per square inch) demonstrating the appearance of wear during rolling friction only under a pressure of 50 kilograms (110 pounds) it was believed

[3]Schulz and Lange, Werkstoffausschussbericht 90 des Vereins Deutscher Eisenhuettenleute, or *Stahl und Eisen*, 1926, p. 1244.

[4]Sitzungsbericht des Verschleissausschusses des Deutschen Verbandes fuer die Materialpruefungen der Technik, Dresden, 1928.

[5]Eden, Rose and Cunningham, Proceedings, Institute of Mechanical Engineers, 1911, p. 875.

[6]Tomlinson, "The Rusting of Steel Surfaces in Contact," *Proceedings*, Royal Society of London, Vol. 115, p. 472 to 483.

[7]French, Rosenberg, Harbaugh and Cross, "Wear and Mechanical Properties of Railroad Bearing Bronzes at Different Temperatures." Forthcoming Bureau of Standards Technologic Paper. *Proceedings*, American Society for Testing Materials, Volume 28, 1928, Part II, p. 298-325. Discussion, p. 348.

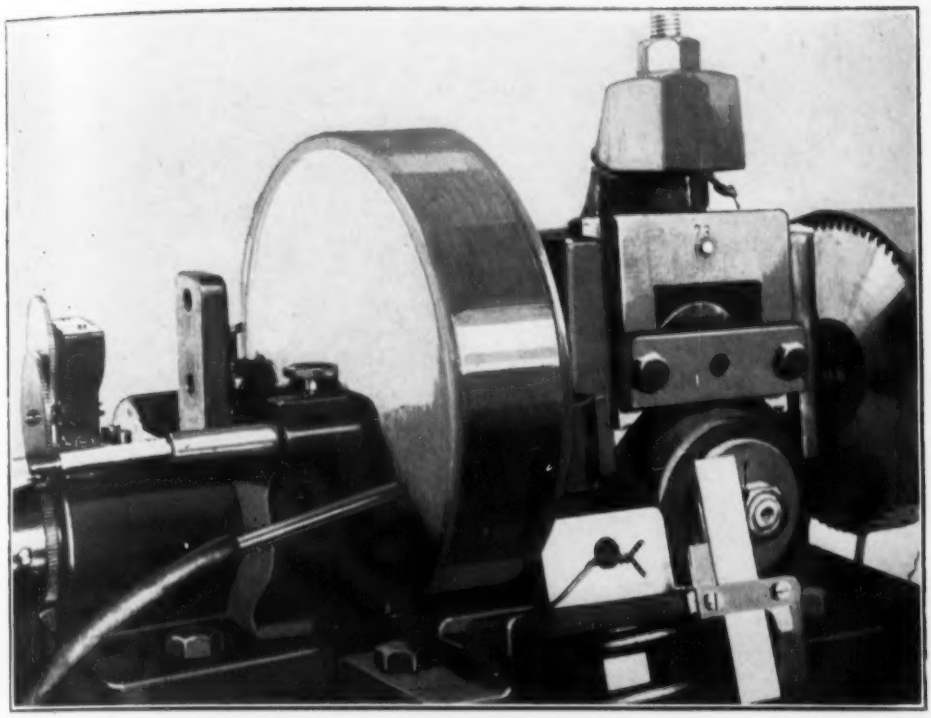


Fig. 2—Gas Cell with Glass Cylinder by which the Wearing Process in Gas May be Observed.

that the oxygen of air had an important influence on the wearing process. To prove this, researches were made on an Amsler wear testing machine with a tire steel with the following analysis and physical properties:

C	Mn	P	S	Si
0.65	0.38	0.03	0.025	0.42

Ultimate Strength
81.8 kilograms per square millimeter (116,220 pounds per square inch)
Elongation
13.8 Per Cent

The pressure between the two test disks was 50 kilograms, sliding 1 per cent and 250 revolutions per minute. In the first series of experiments the wearing process was carried out as usual in air with a loss in weight (one disk) after 50,000 revolutions in one case of 0.1802 grams. In the second series the oxygen of air was eliminated by using an inert gas (nitrogen), carefully purified from oxygen and conducting it in a gas cell (Figs. 2 and 3) surrounding the wear test disks. In this case the loss of weight of the disks under the same

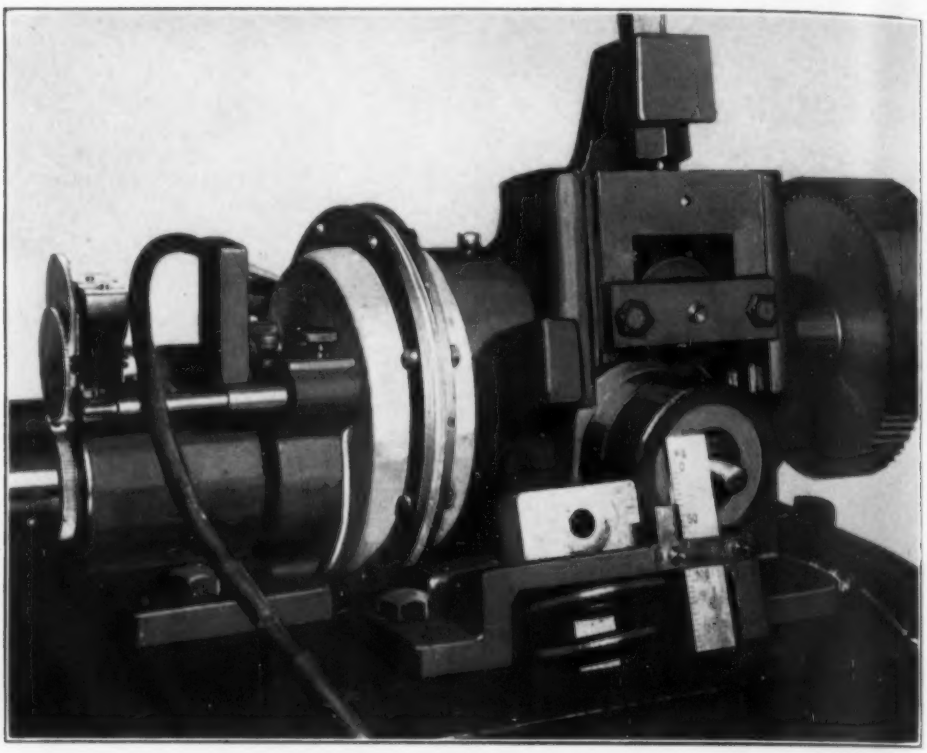


Fig. 3—Gas Cell Completely Closed.

conditions after 50,000 revolutions was 0.0000 grams. No wear took place when oxygen was excluded from the wear test disks.

The oxidation by these results, becomes an essential component of wear. We have then three components of wear:

1. Mechanical removal of particles
2. Cold-hardening process
3. Wear-oxidation.

The participation of these three components in practice to a greater or less extent accounts for the great difference in the wear of materials.

It was possible to observe through the glass cylinder of the first gas cell the upper surface of the wear test disks which became smooth and bright as a mirror during the "wearing" or better, friction process. In Fig. 4 the test disks are seen, the left one being roughened by the wearing process in air, the middle one ground on a grinding machine before the wear test and the right one has the mirror-like

surface after the friction process using 1 per cent sliding friction in an inert gas free from oxygen.

The friction moment which is measured and noted by the tooth-pressure-balance (Zahndruckwaage) Fig. 5 of the Amsler wear testing machine fell to one-third of the value it had been in the air, during the wearing process in an inert gas. It is, therefore, of im-

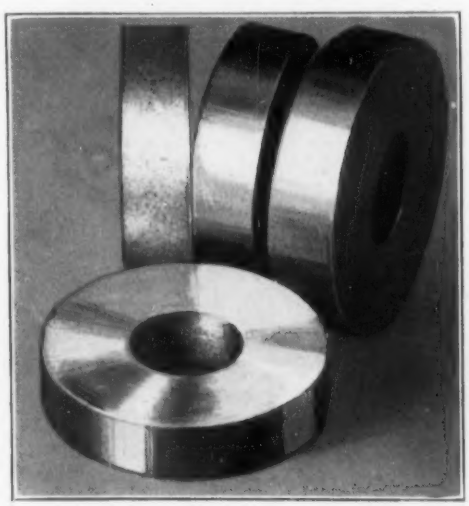


Fig. 4—Four Test Disks. The Left Disk After the Wearing Process in Air, the Middle After Grinding Before Wear Test and the Right After the Wear Test in an Inert Gas Free from Oxygen.

portance to evaluate the value of rolling friction only in an inert gas free from oxygen or directly in a vacuum, because oxygen determines not the friction between metals but the oxidized dust or intermediate substance.

In the first period of wear in air it can be observed that the upper surface grows yellow, red, violet and purple, which are the colors known when steel is heated to a temperature of 200 to 300 degrees Cent. However, the test disks have no higher temperature than room temperature. The wear oxidation produces oxide films on the upper surface which grow thicker and thicker and develop interference to the light.

An interesting fact is that wear with rolling friction begins with an increase in weight; by forming oxide films oxygen is bound to the surface molecules and during this period no wear dust is falling, the weight increases, for instance in one case by 0.0012 grams. The

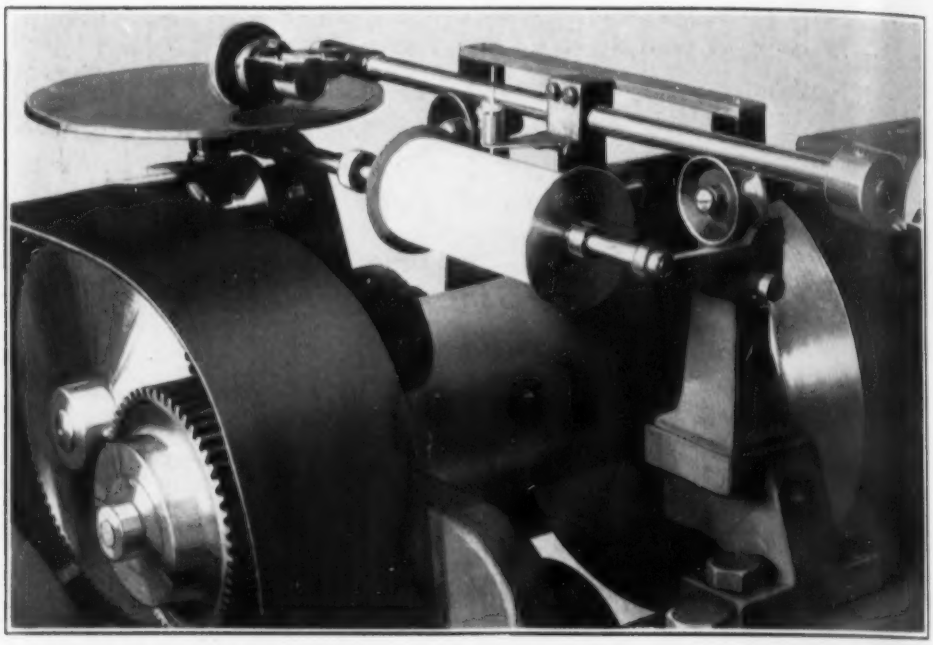


Fig. 5—Amsler Tooth Pressure Balance to Determine the Friction Moment.

wearing curves published hitherto have not taken into account this phenomenon.

After the dark blue color appears on steel disks a yellow-brown film like brown lac appears and it is supposed that this is the lowest iron oxide described by Schenck (Muenster, Germany) who found it in another way to have the formula Fe_3O. When this film grows black, the wear dust begins to fall. The theory by which the oxidation is explained is based on the influence of adsorption. It is a fact that near the surface of metals in any gas there is a higher gas pressure, produced by the free forces of molecules in the surface by which the gas molecules are bound close to the metal molecules. This pressure may be high in some cases (the catalytic effects of some metals are based on this phenomenon.) In the wearing process the surfaces of metals are in contact when forming the well known Hertz surface. In this surface all the small unevennesses are leveled or filled by the disk's pressure and the gas surrounding the metal being already at a higher pressure than atmospheric by the influence of adsorption the result is a further increase of pressure by the contact of metals.

The wear dust, a great part being FeO, is thought to be formed in accordance with the following equation:

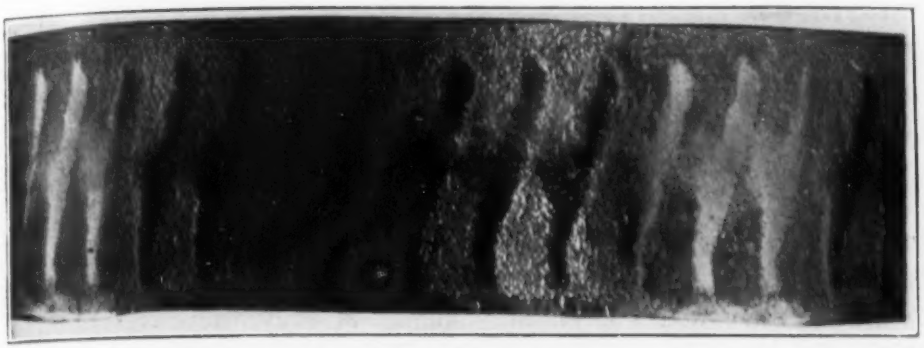


Fig. 6—Corrugation on an Armco Iron Wear Test Disk. The Figures on the Disk which are Sometimes Dark and Sometimes Light are the Places of Less Oxidation. The Oxidized Places are Roughened.

$$2\ Fe + O_2 = 2\ FeO$$

The volumes on the left are the solid Fe and gaseous O_2, on the right the solid FeO. In consequence of Le Chatelier Law an equilibrium is moved by pressure in such a direction that the newly formed products have less volume than the original substances. The equilibrium is moved to the right by application of pressure because the volume of O_2 is greater than the volume of solid FeO. FeO permanently formed during wear (of steel) the process may be continued until all iron is oxidized.

It is of some interest that metals which are known to be very resistive to the influence of oxygen, for instance gold and the Krupp V2A (Cr-Ni Steel) which latter is oxidizing generally not below 800 degrees Cent. are also influenced by wear-oxidation. At the surface of some electro-gilt steel disks it was possible under wearing conditions to wipe off an olive-green colored oxidized wear dust.

Very remarkable is the wear oxidation of the austenitic manganese steel (14 per cent manganese) (Boehler steel works, Duesseldorf). The wear dust here is not quite black but brown. The chemical reduction of the wear dust with H_2 manifests that Fe is oxidized to FeO and manganese to MnO_2; the latter was proved by a paper impregnated with iodine-potassium-iodide starch which became intensely blue, when dipped into a hydrochloric acid solution of the wear dust.

The great resistance of nitrided steel is due to the great hardness and to the impossibility of the iron-nitride oxidizing. In the discussion attached to the article "Hardness of Nitrided Steel" by G. M. Eaton in the TRANSACTIONS of the American Society for Steel

Treating, 1929, No. 1, p. 30, Frederick Franz suggests that ductility cannot be of great influence on wear resistance because diamonds, rubies and sapphires used in watches are very resistant to wear. In these cases the great hardness and non-oxidizability have a much greater importance.

By wear-oxidation corrugation of steel rails can be explained. It has been ascertained by several investigators that vibrations are excited during the rolling of a train over the rails, the cause of the vibrations we will not examine here but only the effect of wear. Vibration produces an alternating pressure and sliding between wheel and rail. Wear-oxidation is greater when greater pressure and sliding occurs between metals and thereby the corrugations or ripples are produced. These corrugations always show bright places with less wear oxidation, and dark places, Fig. 6, where the metal is more oxidized. After wear-oxidation has started the differences between dark and bright places may become of such dimensions that the falling effect of the wheel may deform the structure of the rails.

A protection of the wearing metals with oil does not prevent wear. It is supposed that oxygen which is dissolved in the oil effects wear. By this the wear of gears and bearings working in oil is explained and their tests should be made considering wear-oxidation.

Wear-oxidation is also of much importance during fatigue of metals. The surfaces of fatigue fractures in most cases are oxidized and it is obvious that the fatigue process is influenced by this phenomenon. It is suggested that the friction of surface molecules with the adsorbed oxygen molecules causes this oxidation.

The newly discovered wear-oxidation component, by which the phenomenon of wear is put in one line with other mechanico-chemical processes as corrosion and erosion may solve numerous new problems in the researches of metals.

AN INVESTIGATION OF THE PHENOMENA OF THE STRENGTHENING OF STEEL AND ITS BEHAVIOR UNDER REPEATED OVERSTRESSES

By Professor Dr. A. Thum and Dr. Ing. W. J. Wiss

Abstract

This paper summarizes an investigation on the endurance limit of steels when subjected to dynamic stresses. The importance of this can be realized when it is considered that practically all engineering structures are subjected to these stresses and that materials vary quite widely in their abilities to withstand them. It was found that the endurance limit of certain specimens could be raised by previously stressing them to varying degrees and under varying conditions. The effect of loading and complete unloading, a condition frequently encountered in practice was investigated.

MODERN engineering deals with few structures which are not subjected to repeated stresses (dynamic stresses). Subject to these, metals fail under considerably lower stress than they would under one single (static) stress. Consequently, it is necessary to apply stresses which are far below the yield point of materials to prevent structural failures. Materials show great variations in their abilities to resist repeated stresses. Some, with equal yield points and ultimate strengths, have a high endurance limit, while others have a low one. The aim is to attain an endurance limit as high as possible.

American investigators lately found that in analogy with static stress, material may be strengthened, in certain cases, with repeated (dynamic) stresses. The endurance limit of certain steel specimens was raised by slowly increasing the load while the specimens were subjected to dynamic stressing. The ultimate strength which stood an indefinite number of reversals without failure was called "upper endurance limit" in distinction to the lower or usual endurance limit withstood by the unstrengthened material.

The phenomena of strengthening material by repeated stress (dynamic stress) are a matter of great consequence. A question

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Summary of scientific dissertation submitted by the authors taken from the *Zeitschrift des Vereins Deutscher Ingenieure* constituting the dissertation of Dr. Ing. accepted by the Technical University of Darmstadt, Germany.

closely allied to these phenomena concerns the influence which is exerted by repeated overstresses lasting a short or long time, which may occur within every machine element. The problem is whether they raise or lower the strength of the material.

For the investigation of these questions experiments were made to determine the structural changes accompanying strengthening of steel. The metallographic methods employed for this purpose did not show any modification of structure which would have been pro-

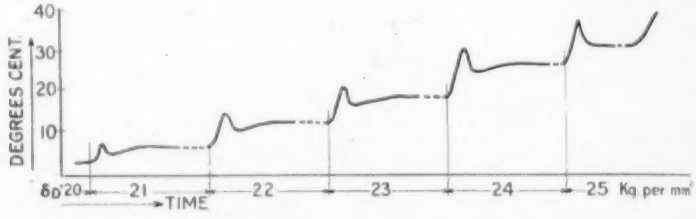


Fig. 1—Curve for Long Endurance Test. Increase of Load 1 Kilogram per Square Millimeter After Each 5,000,000 Cycles.

duced by the strengthening cycles applied. These investigations did not produce satisfactory results and were abandoned in favor of later metallographic developments and X-ray experiments.

The authors studied modifications in the materials by means of observing temperature, consumed energy and deflection. The investigations were made on a Schenck rotating-beam testing machine, the first experiments were with a soft steel (0.14 per cent carbon; 0.49 per cent manganese; 0.03 per cent phosphorus; 0.05 per cent sulphur; ultimate strength 54,600 pounds per square inch; yield point 33,400 pounds per square inch; reduction in area 34.3 per cent). The principal results were checked afterwards by specimens of other materials.

The investigations started with the "long-endurance test." Beginning with the endurance limit, specimens were subjected to a stress which was advanced 1400 pounds per square inch after each 5 million cycles. The temperature first rose a few degrees but decreased after a short time; it then increased again very slowly with the tendency to a stationary value (shown in Fig. 1). This reoccurred with each increase of load.

The results show that the endurance limit, which with an immediate application of load is low, is, under certain conditions, raised considerably. For example, in the low carbon steel the endurance limit was increased from 28,400 to 34,100 pounds per square inch,

i. e. by 20 per cent. A medium carbon steel (0.50 per cent carbon; ultimate strength 95,800 pounds per square inch; yield point 63,400 pounds per square inch; reduction of area 17 per cent) had the endurance limit increased from 48,300 to 52,600 pounds per square inch. The results showed that at least 10 million cycles must be applied and that the test should start from below the usual endurance limit if all capacity of strengthening is to be exploited.

The next problem concerned strengthening in shorter intervals. Stress was increased 1400 pounds per square inch after each 50,000 and less cycles. Alternating the number of stressed cycles in different

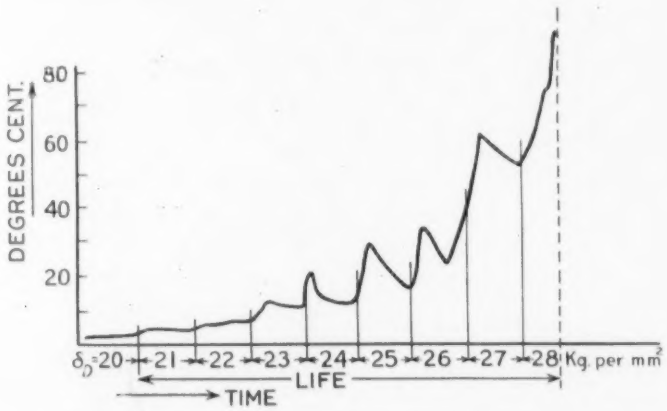


Fig. 2—Temperature Curve for Short Endurance Test on Low Carbon Steel. Increase of Load 1 Kilogram per Square Millimeter After Each 100,000 Cycles.

manners gave an indication of the influence of the speed of strengthening on the life and the ultimate strength. In this connection life is designated as the number of cycles which the specimen withstands from the beginning of the test until failure (see Fig. 2). The more slowly stress is increased, the more the life is increased but the more rapidly ultimate strength is decreased. After each increase of stress, unless it took place too fast, the temperature and energy produced by bending strains decreased. First there was sudden ascent, then oscillation and finally a constant value was reached. This oscillation in the process became particularly marked, starting from the range of "upper endurance limit" (see Fig. 2).

Material was more quickly strengthened beneath the range of increased strength. Since the complete strengthening requires a great many cycles, life was increased by the number of cycles spent during the strengthening process. A higher ultimate strength resulted by

increasing the stress more rapidly. A specimen on which the load was increased after each 10,000 cycles attained a life of 0.42×10^6 cycles and an ultimate strength of 43,000 pounds per square inch, another one on which the load was increased after each 0.45×10^6 cycles a life of 1.47×10^6 cycles and an ultimate strength of 41,100 pounds per square inch.

If the specimens in the pre-stressed state were tested by increasing the load, a long life could not be obtained. When the test was

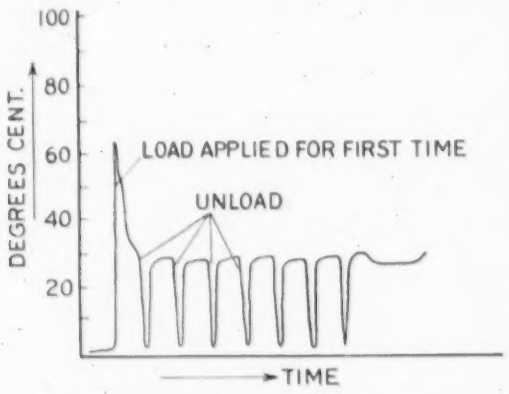


Fig. 3—Temperature Curve for Repeated Constant Overload Interrupted by Idle Periods.

run to above the endurance limit, the specimen was fatigued and a shorter life than with an unfatigued specimen was obtained. Unfatigued material is distinguished by the greatest life when exposed to rising load.

If the specimen is subjected to repeated constant overloads alternating with complete unloading (shown in Fig. 3) its life is decreased. This kind of straining occurs often in practice in the sudden strains applied to vehicles or frequently starting hoists. The test results indicate that life, if calculated without the idle cycles, is diminished since the strengthening process has been interrupted. The same results that were obtained with continuous straining, namely, increase of temperature and energy to a maximum closely followed by a decrease, were obtained with a continually rising stress periodically interrupted by unloading (Fig. 4). From this the authors argue that every short overloading has its exact values of temperature and energy which take place immediately after applying the load, and which are modified only by a very small amount with short unloading.

In another part of the investigation the specimens were strained

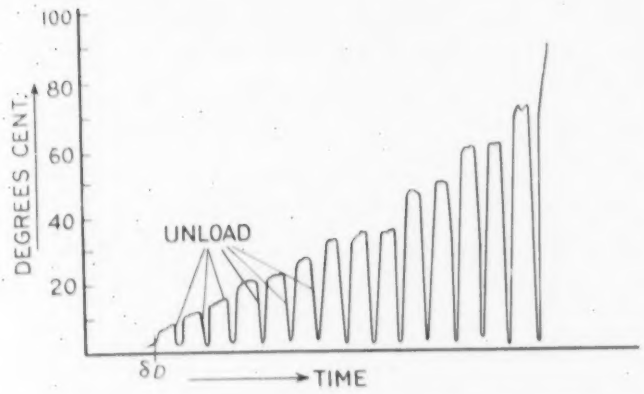


Fig. 4—Temperature Curve for Repeated, Increasing Overload Interrupted by Idle Periods. Increase of Load 4 Kilograms per Square Millimeter After Each Period.

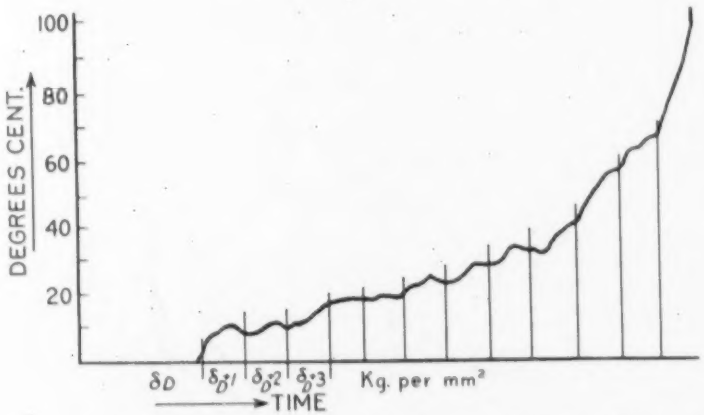


Fig. 5—Temperature Curve for Short Endurance Test on Tool Steel. Increase of Load 1 Kilogram per Square Millimeter After Each 75,000 Cycles. (Compare with Fig. 2.)

until failure and the increases of temperature and energy (method of Woehler and the University of Illinois) were observed. The results show that a complete retrogression of these values took place a short distance below the upper endurance limit after the increase of load had been worked up; there was no more retrogression but temperature and energy increased continuously until failure. At that point the materials had passed over from the elastic to the plastic range of strength in analogy with the plastic state with static strength.

The next experiments concerned the behavior of specimens if subjected first to a limited number of cycles and afterwards to the endurance limit. The tests showed that a limited overstress can in certain cases quite well be withstood. For example, a specimen of the low carbon steel (endurance limit 28,400 pounds per square inch)

did not fail when tested with 200,000 cycles and 37,000 pounds per square inch stress, or 100,000 cycles and 35,000 pounds per square inch stress, or 50,000 cycles and 34,100 pounds per square inch; it continued running practically unlimited after the endurance limit had been applied to the specimen.

From the other materials tested there ought to be mentioned especially a tool steel (silver steel, with 1.2 per cent carbon, 0.38 per cent tungsten, 0.33 per cent manganese, 0.26 per cent silicon) and chromium-nickel steel (0.38 per cent carbon, 0.76 per cent manganese, 0.21 per cent silicon, 1.75 per cent nickel, 0.8 per cent chromium). These specimens had a great deal smaller increase of temperature and energy and a correspondingly smaller decrease after applying the overstress than the low carbon steel.

It is of interest to know whether material already strengthened in a static way can be strengthened more by the dynamic method. As shown by the results the endurance limit increased by applying this method of straining; but further dynamic strengthening is scarcely to be attained because the specimen has already consumed its strengthening capacity during the first strain.

The influence which artificial seasoning of cold-drawn material takes relative to the endurance strength was investigated. Here the authors observed that the endurance limit was diminished by only a small amount of seasoning and the effect of the cold drawing was, therefore, decreased slightly.

The investigations also allow some conclusions concerning the theories of the fatigue of metals. It should be emphasized that strengthening by repeated stresses always consumes elasticity and produces brittleness. By absorbing the dampening the specimen becomes very delicate to overstress. The continuous inner reversed strain consumes the cohesion strain in such a way that minute localized flaws of less strength are produced which finally cause failure.

FATIGUE TESTS OF SMALL SPECIMENS WITH PARTICULAR REFERENCE TO SIZE EFFECT

By R. E. Peterson

Abstract

This paper gives data concerning a small fatigue testing machine weighing ten pounds and taking a test specimen fifty thousandths of an inch in diameter at the critical section. Fatigue tests on this small machine are compared with similar tests on larger machines. It is shown that for ordinary steels no appreciable size effect occurs up to two inch diameter. For cast iron the results obtained with small specimens are erratic and considerably lower than for specimens of ordinary size. Some observations are also made on heating effect in specimens of different diameters.

Introduction

THE reasons for the development of a machine for testing small specimens were two in number: 1. to enable fatigue tests to be made where only a small amount of material is available, such as in broken machine parts, screws, turbine blades, etc.; 2. to study size effect. One theory of fatigue failure is based on surface defects[1] and, if true, some change in endurance limit would be expected in testing various sizes of specimens, due to the fact that the stress concentration caused by a small discontinuity is theoretically[2, 3, 4] a function of d/D, the ratio of size of flaw to size of specimen. The question of size effect is of practical importance to engineers, who are generally sceptical in applying data from "lead pencil size" test specimens

[1]Griffith, "Phenomena of Rupture and Flow in Solids", *Philosophical Transactions*, Royal Society, Vol. 221 A, 1920, p. 163.

[2]Timoshenko and Dietz, "Stress Concentration Due to Holes and Fillets", *Transactions*, American Society of Mechanical Engineers, 1926, p. 199.

[3]Wewerka, "Die Kerbwerke," *Maschinenbau*, Jan. 17, 1929.

[4]Peterson, "An Investigation of Stress Concentration", *Mechanical Engineering*, Dec. 1926, p. 1449.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, R. E. Peterson, is associated with the mechanics division research laboratories of the Westinghouse Electric and Manufacturing Co., East Pittsburgh. Manuscript received June 16, 1930.

to the design of large machine parts, such for example as generator shafts thirty inches in diameter.

Description of Small Fatigue Testing Machine

The small fatigue testing machine (Fig. 1) is of the rotating cantilever type, the load being applied directly by means of weights. The size of specimen used is fifty thousandths of an inch in diameter

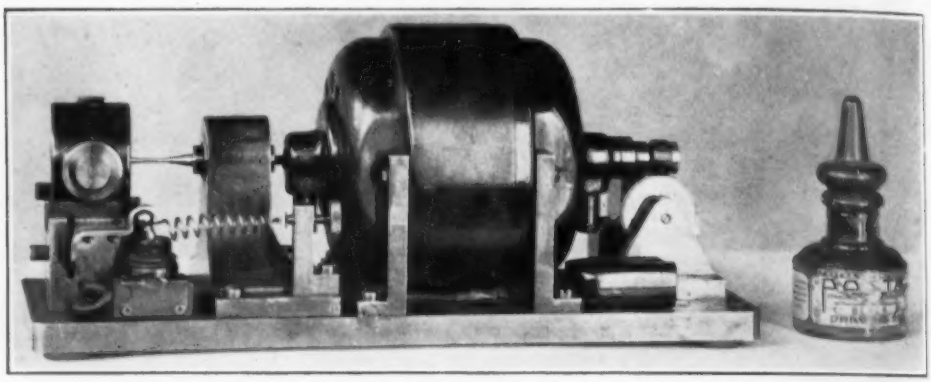


Fig. 1—Small Fatigue Testing Machine.

at the critical section, which is formed by turning on a contour radius of two inches. The relative size of this small specimen as compared with a 2¼-inch diameter specimen[5] can readily be visualized. The machine is driven by a standard fan motor, operated by plugging into an ordinary light socket. The total weight of the machine is slightly less than 10 pounds, and when equipped with a carrying case is as portable as an ordinary voltmeter. The changing of specimens is a quick operation inasmuch as the specimen fits directly into three small ball bearings, with no sockets or sleeves. The usual counter and arrangement for tripping a switch at failure are provided as shown in Fig. 1.

Size Effect

Tests were made of sizes as follows:

1. 0.050 inch diameter—Small machine described in this paper.
2. 0.273 inch diameter—Sondericker-Farmer type machine.[6]

[5]Peterson, "Fatigue Tests of Large Specimens", *Proceedings*, American Society for Testing Materials, Vol. 29, Part II, 1929, p. 371.

[6]Moore and Kommers, "Fatigue of Metals", McGraw-Hill, p. 94-97.

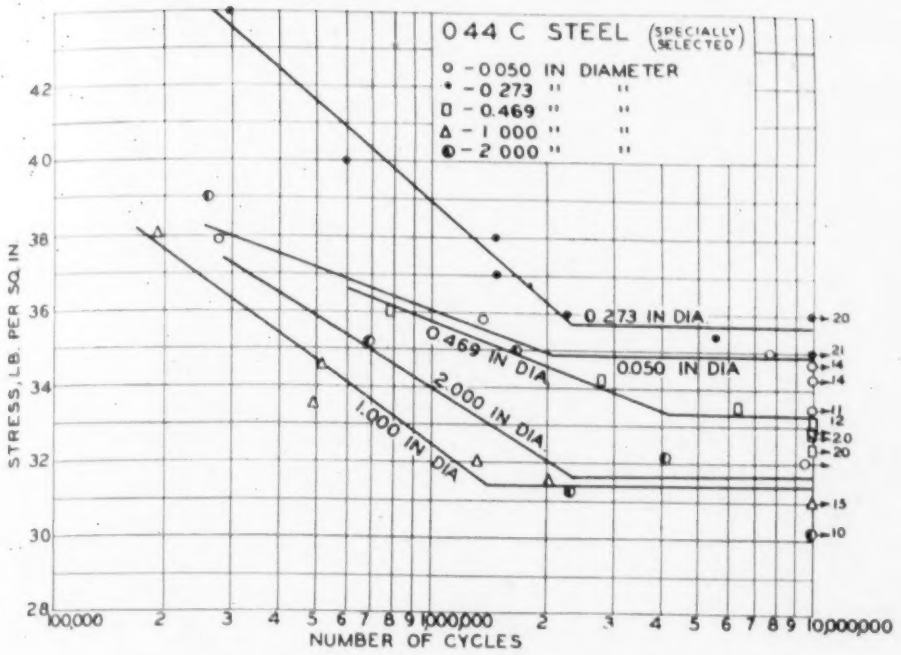


Fig. 2—Curves Showing Results of Size Effect.

3. 0.469 inch diameter—Olsen machine using McAdam tapered cantilever specimen.[6]
4. 1.000 inch diameter—Machine described in previous paper.[5]
5. 2.000 inch diameter.[5]

A photograph of the above specimens (with exception of 0.050 inch diameter) is also shown in the paper mentioned.[5]

The materials tested were the following:

1. Medium carbon steel (specially selected).

This steel was especially purchased for these tests, with specifications requiring that the material be uniform and clean. Numerous photomicrographs were taken to check uniformity. Analysis showed 0.44 per cent carbon, 0.64 per cent manganese. The physical properties were: yield point, 43,700 pounds per square inch; ultimate strength, 81,700 pounds per square inch; elongation, 32 per cent; reduction of area, 50 per cent.

2. Medium carbon steel (ordinary hot-rolled bar stock).

This material was taken from 1½-inch diameter hot-rolled bar stock which as a rule is none too uniform and is sometimes quite "dirty". Analysis showed 0.42 per cent carbon, 0.62 per cent manganese, 0.15 per cent silicon, 0.028 per cent sulphur, 0.017 per cent phosphorus. The physical properties were: yield point—37,200

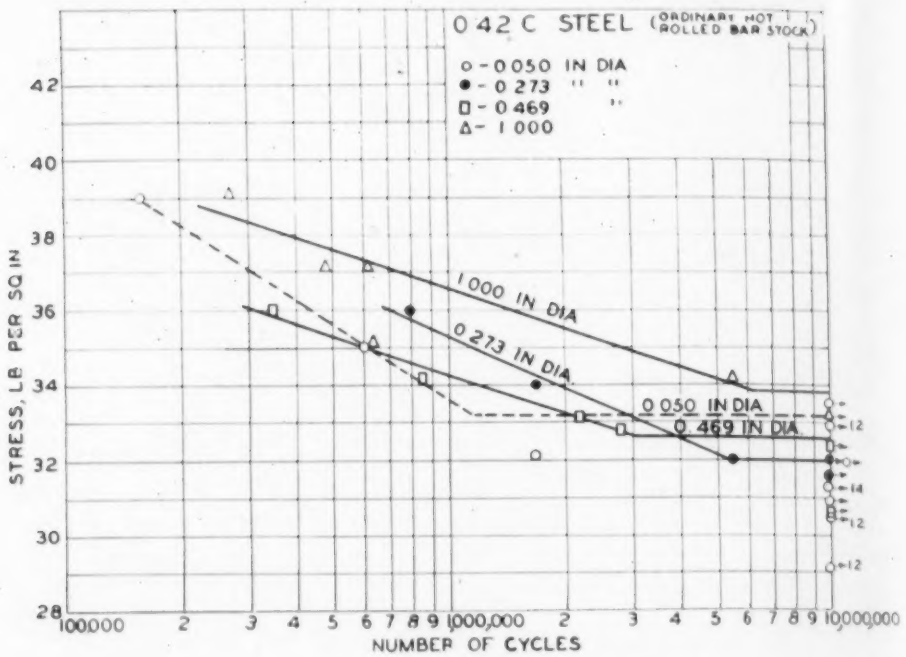


Fig. 3—Curves Showing Results of Size Effect.

pounds per square inch; ultimate strength, 73,800 pounds per square inch; elongation, 34 per cent; reduction of area, 51 per cent.

3. Rotor steel (heat treated).

A description of this material, which is used in large rotor forgings, has been published.[7] Analysis showed 0.57 per cent carbon, 0.60 per cent manganese, 0.26 per cent silicon, 0.039 per cent sulphur, 0.037 per cent phosphorus. The heat treatment was: 1600 degrees Fahr., 5 hours, air cool; 1300 degrees Fahr., 5 hours, furnace cool; 1490 degrees Fahr., 5 hours, water quench; 1200 degrees Fahr., 4 hours, furnace cool. The physical properties were: yield point, 67,000 pounds per square inch; ultimate strength, 102,000 pounds per square inch; elongation, 20 per cent; reduction of area, 41 per cent.

4. Cast iron.

The test pieces were cut from a large gray iron casting, all pieces being taken at the same distance from the surface of the casting. The cast iron was of a good grade, containing no large holes or flaws.

The results on size effect are given in Tables I to IV and

[7] Merten, "High Temperature Treatments of Castings and Forgings as Evidenced by Core Drill Tests from Heavy Sections", TRANSACTIONS, American Society for Steel Treating, Jan. 1928.

Table 1
Fatigue Test Data
0.44 Per Cent Carbon Steel (Specially Selected)

No.	Diam. of Specimen Inches	Stress Lbs. Per Sq. In.	Number of Cycles	Notes
S-2	0.050	35,000	1,668,000	
S-6		32,100	9,488,000	No failure.
S-7		41,200	62,000	
S-8		37,900	280,000	
S-9		35,800	1,357,000	Slight vibration.
S-10		32,900	9,924,000	No failure.
S-15		33,500	11,808,000	No failure.
S-17		34,300	14,171,000	No failure.
S-18		35,000	7,691,000	
S-19		34,700	13,913,000	No failure.
		34,900		Endurance Limit.
	Inches			
B-1	0.273	44,000	296,000	
B-2		40,000	602,000	
B-3		36,000	20,453,000	No failure.
B-4		28,000	1,482,000	
B-5		37,000	1,488,000	
B-6		36,000	2,257,000	
B-7		35,000	21,604,000	No failure.
B-8		35,400	5,280,000	
		35,700		Endurance Limit.
	Inches			
C-1	0.469	36,000	780,000	
C-2		32,400	20,000,000	No failure.
C-3		34,200	2,790,000	
C-4		33,500	6,378,000	
C-5		32,760	20,000,000	No failure.
C-6		33,120	12,000,000	
		33,300		Endurance Limit.
	Inches			
139-1	1.000	33,500	493,000	Slight coloring. Broke outside colored zone.
139-2		34,500	518,000	Blue color. Broke at critical section.
140-1		32,000	1,326,000	No color. Broke at critical section.
140-2		31,000	14,716,000	No color. No fracture.
144-1		38,000	192,000	Deep blue. Broke outside colored zone.
144-2		31,500	2,060,000	No color. Broke at critical section.
		31,400		Endurance Limit.
	Inches			
137	2.000	35,200	694,000	Deep blue. Broke outside colored zone.
138		32,200	4,129,000	No color. Failure away from critical sec.
143		31,300	2,299,000	No color. Failure away from critical sec.
146		30,200	10,000,000	No color. No failure.
147		39,200	265,000	Deep blue. Broke outside of colored zone.
		31,700		Endurance Limit.

shown graphically in Figs. 2 to 5. The endurance limits obtained are plotted in a "size effect" diagram (Fig. 6). The endurance limits obtained for 0.57 per cent carbon steel (heat treated) are practically the same for 0.050-inch diameter and for 0.469-inch diameter. For the medium carbon steels, a line corresponding to an average endurance limit of 33,500 pounds per square inch represents the data fairly well, the maximum deviation being 6 per cent.

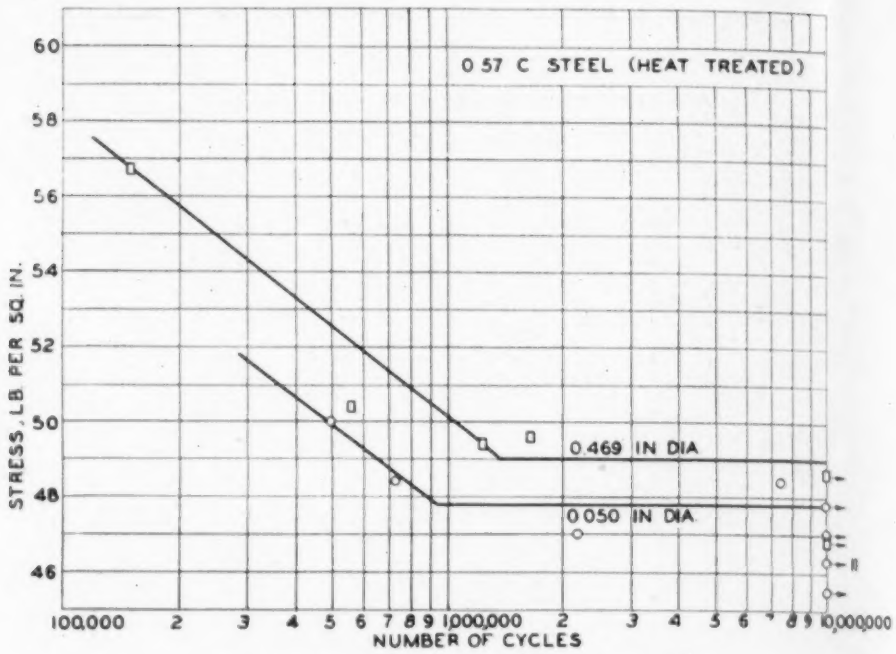


Fig. 4—Curves Showing Results of Size Effect.

It seems therefore that for ordinary steels the endurance limit is independent of size. A general statistical effect seems to apply even at a diameter of fifty thousandths of an inch. It is probably necessary to decrease in size to something approaching grain size in order to obtain a noticeable size effect.

It will be noticed from Fig. 6 that in the tests of medium carbon steel, the specially selected steel did not give test results of greater uniformity than the ordinary hot-rolled bar stock. This does not mean that one need not be careful in purchasing steel, particularly since considerable evidence is available which indicates relatively wide "scatter" of data for dirty material.[8]

For cast iron, a considerable reduction of endurance limit was obtained with small specimens (26 per cent). The wide "scatter" of test data obtained from small cast iron specimens is shown in Fig. 5. The fractures occurred in different places on the test pieces, depending apparently on the distribution of the tiny holes on the surface. From these facts, it may be concluded that small specimens should not be used for testing the fatigue properties of porous materials, such as cast iron, weld metal, etc.[5]

[5]Gillett and Mack, "Molybdenum, Cerium and Related Alloy Steels", Chemical Catalogue Co., p. 120, 148.

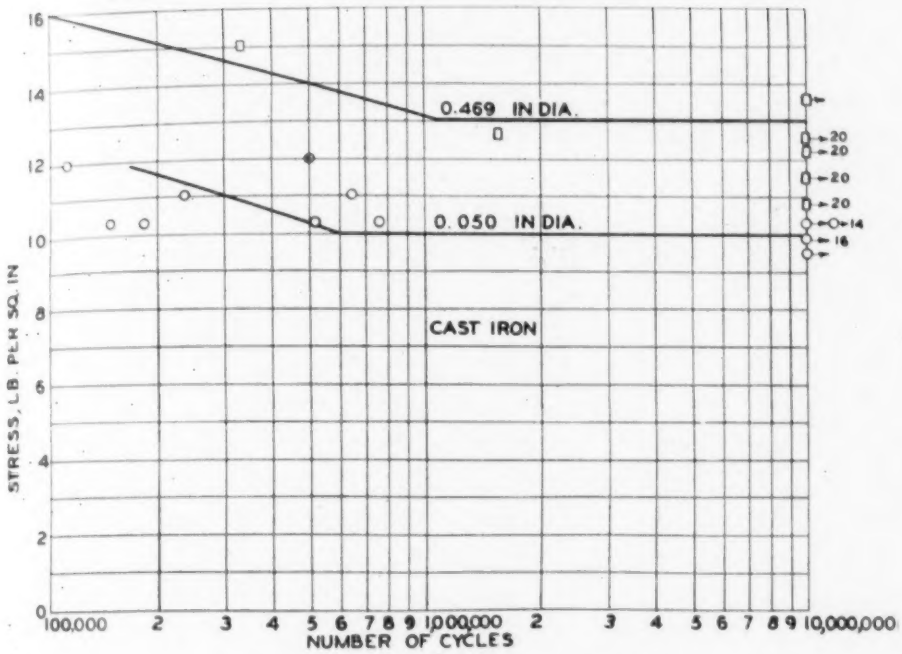


Fig. 5—Curves Showing Results of Size Effect.

An interesting observation was made in testing specimens of various diameters. It was noticed that if the stress was somewhat above the endurance limit, the large specimens (1 and 2 inches in diameter) became very hot, coloring in the region of the critical section to deep blue (corresponding to about 600 degrees Fahr.).[9] Heating of this kind is, of course, due to internal friction. A heating factor representing heat generated (some function of volume) to heat dissipated (some function of surface) may be set up as follows:

$$c = \frac{k_1 V}{k_2 S} = \frac{k_1 \frac{\pi}{4} d^2 l}{k_2 \pi d l} = kd$$

The heating effect is therefore greater for the large test specimens. In these tests, the limit for a heating effect sufficient to cause coloring with application of stress somewhat above the endurance limit corresponds to a diameter between ½ and 1 inch.

It was also observed that when coloring took place, fracture occurred outside of the colored region (Fig. 7), which seems to

[9]Bullens, "Steel and Its Heat Treatment", John Wiley and Sons, p. 192.

Table II
Fatigue Test Data
0.42 Per Cent Carbon Steel (Ordinary Hot Rolled Bar Stock)

No.	Diam. of Specimen Inches	Stress Lbs. Per Sq. In.	Number of Cycles	Notes
S-1	0.050	39,000	158,000	Vibration.
S-3		35,000	602,000	
S-4		32,100	1,668,000	Slight vibration.
S-5		29.100	11,851,000	No failure.
S-12		30,500	11,726,000	No failure.
S-20		30,900	11,384,000	No failure.
S-21		31,300	14,273,000	No failure.
S-22		32,100	10,052,000	No failure.
S-24		32,900	11,847,000	No failure.
S-25		33,500	9,928,000	No failure.
		33,200		Endurance Limit.
	Inches			
B-1	0.273	36,000	800,000	
B-2		34,000	1,700,000	
B-3		32,000	5,480,000	
B-4		32,000	20,000,000	No failure.
B-5		31,600	20,000,000	No failure.
		32,000		Endurance Limit.
	Inches			
C-1	0.469	39,600	50,000	
C-2		36,000	350,000	
C-3		34,200	860,000	
C-4		33,100	2,180,000	
C-5		32,800	2,800,000	
C-6		32,400	20,000,000	No failure.
C-7		30,600	20,000,000	No failure.
		32,600		Endurance Limit.
	Inches			
75-1	1.000	39,150	272,000	Blue. Broke outside colored zone.
75-2		37,150	624,000	Straw color. Broke outside colored zone.
76-1		35,200	642,000	Straw color. Broke outside colored zone.
76-2		33,200	10,231,000	No failure.
77-1		37,150	478,000	Blue. Broke outside colored zone.
77-2		34,200	5,537,000	Lightly colored. Broke at critical section.
		33,800		Endurance Limit.

indicate that for medium carbon steels the endurance limit increases* with temperature[10] up to at least 600 degrees Fahr. A similar phenomenon has been found in tensile tests,[11] and from the correlation between endurance limit and ultimate strength,[12] it is possible to explain the above result.

It should be pointed out that this heating effect has apparently no bearing on the values of endurance limit for large specimens,

*See Appendix A.

[10]Moore and Kommers, ibid, p. 156.

[11]McVetty and Mochel, "The Tensile Properties of Stainless Iron and Other Alloys at Elevated Temperatures", TRANSACTIONS, American Society for Steel Treating, Vol. 11, Jan. 1927, p. 78.

[12]Moore and Jasper, University of Illinois Experimental Station Bulletin No. 136, p. 53.

Table III
Fatigue Test Data
0.57 Per Cent Carbon Steel (Heat Treated)

No.	Diam. of Specimen Inches	Stress Lbs. Per Sq. In.	Number of Cycles	Notes
S-26	0.050	50,000	500,000	
S-27		48,400	738,000	
S-30		45,500	10,320,000	No failure.
S-31		47,000	2,192,000	
S-32		46,300	11,255,000	No failure.
S-33		47,000	10,000,000	No failure.
S-34		47,800	10,170,000	No failure.
S-35		48,400	7,737,000	
		47,800		Endurance Limit.
	Inches			
C-1	0.469	63,700	45,000	
C-2		56,500	150,000	
C-3		50,400	560,000	
C-4		49,600	1,630,000	
C-5		49,400	1,220,000	
C-6		48,600	20,000,000	No fracture.
C-7		46,800	20,000,000	No fracture.
		49,000		Endurance Limit.

Table IV
Fatigue Test Data
Cast Iron

No.	Diam. of Specimen Inches	Stress Lbs. Per Sq. In.	Number of Cycles	Notes
S-36	0.080	11,000	239,000	
S-37		11,000	650,000	
S-38		9,500	11,884,000	No failure.
S-39		10,300	525,000	
S-40		10,300	187,000	
S-41		10,300	10,162,000	No failure.
S-42		9,900	16,005,000	No failure.
S-43		10,300	149,000	
S-44		10,300	770,000	
S-45		10,300	10,000,000	No failure.
S-46		12,000	111,000	
S-47		12,000	500,000	
		10,000		Endurance Limit.
	Inches			
C-1	0.469	16,200	23,000	
C-2		12,600	1,577,000	
C-3		10,800	20,000,000	No failure.
C-4		11,520	20,000,000	No failure.
C-5		12,240	20,000,000	No failure.
C-6		12,600	20,000,000	No failure.
C-7		13,680	10,000,000	No failure.
C-8		15,120	329,000	
		13,000		Endurance Limit.

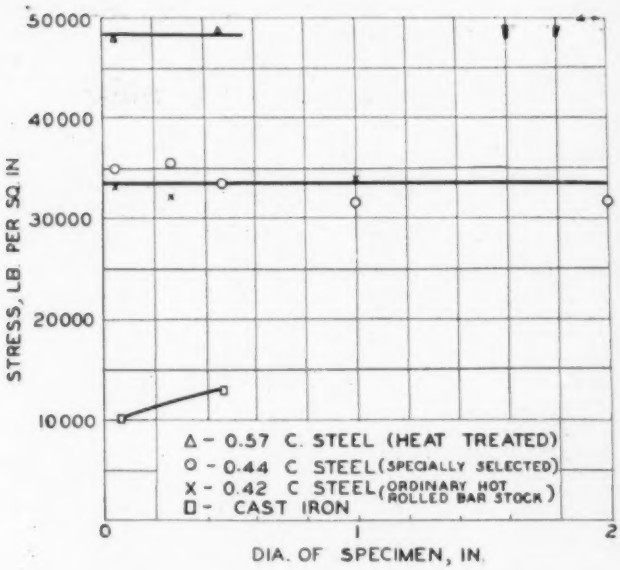


Fig. 6—Size Effect Diagram.

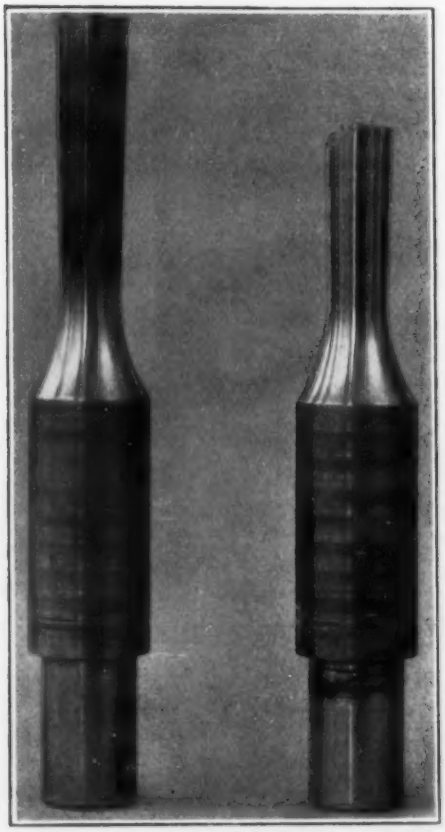


Fig. 7—Photograph Illustrating Shifting of Fracture Due to Heating.

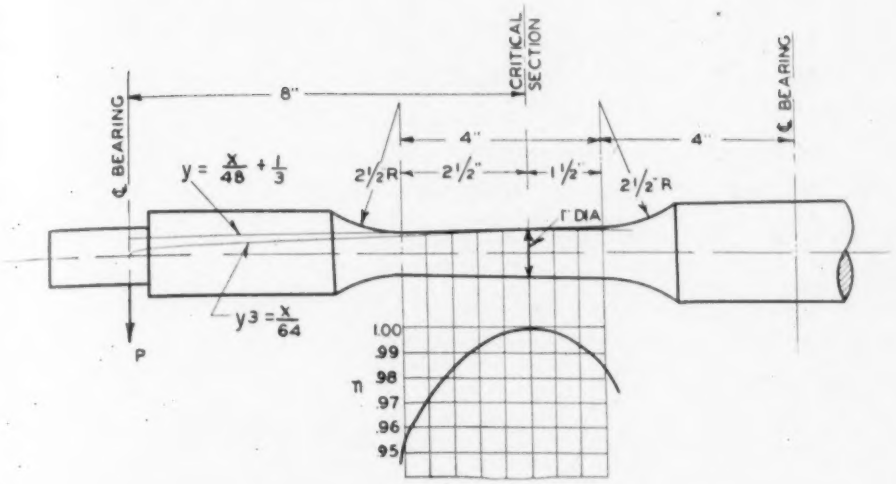


Fig. 8—Stress Distribution in One-Inch Cantilever Fatigue Specimen.

since for stresses slightly above the endurance limit, no coloring took place and fracture occurred at the critical section as in tests of small specimens.

Conclusions

1. A small fatigue testing machine, weighing about ten pounds and using a specimen fifty thousandths of an inch at the critical section, has been developed to a stage where it can be used in making tests where only a small amount of material is available, such as in broken machine parts, screws, turbine blades, etc.

2. No appreciable size effect was found in fatigue tests of carbon steels (0.42 to 0.44 per cent carbon) for sizes 0.050-inch diameter to 2 inches diameter, and 0.57 per cent carbon steel (heat treated) for sizes 0.050-inch diameter to 0.469-inch diameter.

3. For 0.050-inch diameter cast iron specimens, a considerably lower endurance limit was obtained (26 per cent), with wide "scattering" of test data, which makes it inadvisable to use small specimens in tests of porous material such as a cast iron, weld metal, etc.

4. For large specimens (1 and 2 inches diameter) of medium carbon steel stressed somewhat above the endurance limit, heating sufficient to cause coloring occurs. With such a condition, fracture occurs outside of the colored region, indicating for medium carbon steel an increase of endurance limit with increased temperature up to at least 600 degrees Fahr.

Acknowledgments

The writer is indebted to T. F. Hengstenberg for testing of the medium sized fatigue specimens and to F. C. Cassel for operation of the large fatigue testing equipment. The selection of the uniform medium carbon steels was made on the basis of a metallographic investigation by Dr. F. R. Hensel. Particular acknowledgment is due John M. Lessells, manager of mechanics division of the Westinghouse Research Laboratories, for his encouragement and suggestions concerning the development of the equipment and execution of tests.

Appendix A

In order to fully understand the discussion concerning the location of fracture with heating effect, it is necessary to know the stress distribution along the cantilever specimen. A constant stress cantilever beam of circular cross-section has the form

$$S = \frac{4Px}{\pi y^3} \qquad y^3 = kx$$

The slope at any point is

$$\frac{dy}{dx} = \frac{k^{1/3}}{3x^{2/3}}$$

The critical section of a cantilever fatigue specimen is defined by making a tapered surface tangent to a beam of constant stress (Fig. 8). This condition is satisfied in a 1-inch diameter specimen of 8 inch moment arm when the taper is ½ inch per foot on the diameter. $\left(\frac{dy}{dx} = \frac{1}{48}\right)$. The ratio of the stress at any distance x to the stress at the critical section will always be less than unity. For the tapered portion of the above specimen, this ratio becomes

$$n = \frac{S_x}{S_c} = \frac{x}{64} \Big/ \left(\frac{x}{48} + \frac{1}{3}\right)^3$$

A curve plotted from the above equation is shown in Fig. 8. The stress variation is but 1 per cent for 2½ inches along the

tapered portion of the specimen near the critical section. The value of n decreases very rapidly along the portions of the specimen turned with a 2½-inch contour radius.

DISCUSSION

Written Discussion: By H. F. Moore, professor of engineering materials, University of Illinois, Urbana, Illinois.

Mr. Peterson has made a very helpful contribution to the literature of the technique of testing metals under repeated stress. His tentative conclusion that specimens as small as 0.05 inch in critical diameter can be used to

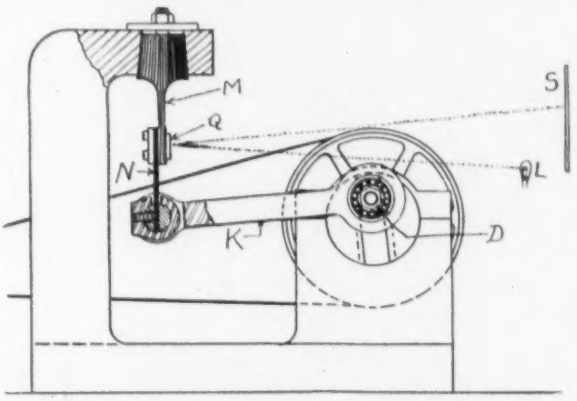


Fig. 1

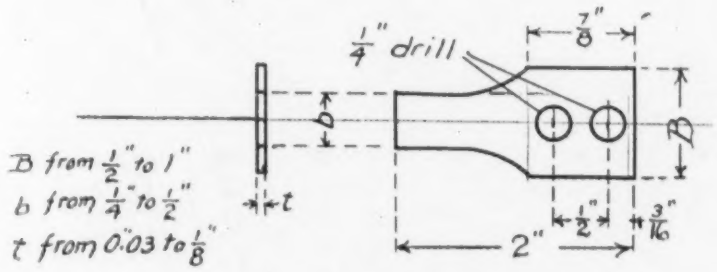


Fig. 2

get the endurance limit of a metal is one which will be very useful to testing engineers, especially if it is confirmed by tests on other metals than steel. Both specimens and testing machines for fatigue tests will be made less expensive if we find we can use such small specimens and can get reliable results from them.

A form of testing machine for small specimens which for several years has been used in the Fatigue of Metals Laboratory at the University of Illinois is shown in Fig. 1. This machine was designed for testing specimens of thin sheet metal, but has proven useful in testing the varying fatigue strength of different layers of rail joint subjected to surface indentation from rail ends.

The specimen *N* is fastened at one end to the calibrated flat spring *M*, and the other end of the specimen is vibrated back and forth by the connect-

ing rod K, which is operated by the variable-throw crank D. If the throw of the crank is increased, the bending moment on the specimen is increased, and the deflection of Q, a mirror attached to the calibrated spring, is also increased, causing motion of a beam of light reflected from the lamp L to the screen S. There is provided an automatic trip which is operated by dropping of connecting rod K when the specimen breaks, and which opens the motor switch, thus stopping the machine. A counter is also provided which records the number of rovolutions.

By the use of shims behind the specimen N the range of stress in the specimen can be altered from complete reversal to one-direction flexure.

The specimen used is shown in detail in Fig. 2.

CHAIRMAN H. W. GILLET:[1] The Westinghouse people are certainly right up to date with their Tom Thumb endurance testing machine. The comments on heating up are very interesting. We have been working on some austenitic stainless steels of the usual 18-8 type and those will heat up if stressed only a little above the endurance limit in the ordinary quarter-inch critical sections or thereabouts. That factor will be a function not only of the size but of the nature and thermal conductivity of the specimens.

T. D. LYNCH:[2] May I have put on the screen again Mr. Peterson's slide illustrating the two extreme sizes of the test pieces used by him; namely, 0.05-inch diameter and 2-inch diameter.

From the position where I was in the rear of the room, I wondered if the significance of the difference of the sections of these two sizes of samples was really appreciated. Those of us who have had the pleasure of associating with Mr. Peterson in these tests have long realized the need for a better knowledge of size effect on the fatigue values of metals.

The small test piece is necessary in testing sections not of sufficient size to use a standard fatigue test bar, and the larger section has been found equally useful in studying welded joints of considerable size. Testing engineers in general realize the importance of knowing as nearly as possible the real value of a material in the size and under the condition of service. Therefore, I wish to emphasize the significance of this knowledge of the relative values of metals of varying cross sections.

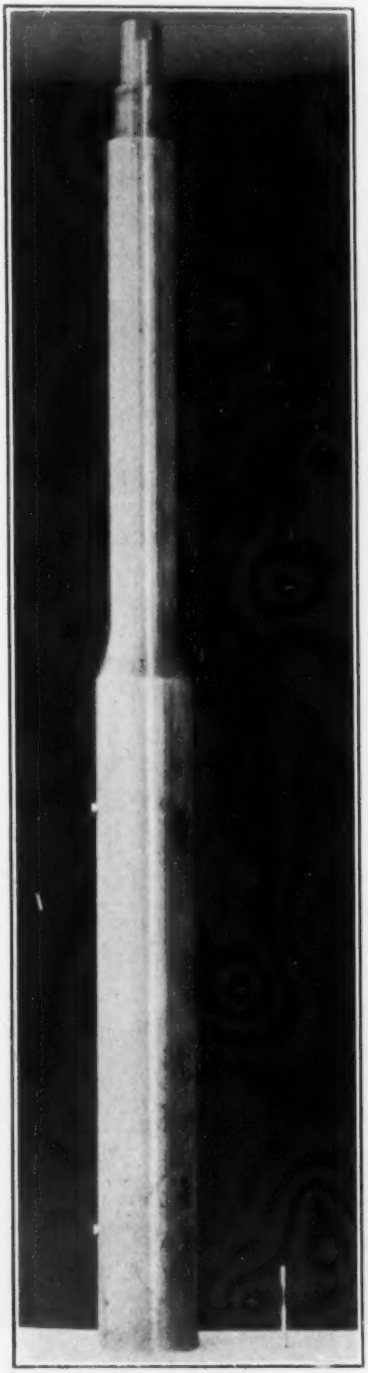
R. L. TEMPLIN:[3] We, like many other investigators, are very much interested in the fatigue or endurance tests of metals, more particularly aluminum and its alloys. We are just starting our tests to determine the size effect of the specimens and it is a little early to attempt to give any detailed comments of what we are finding, but we have received definite evidence that the size of the specimen has a very material influence on results obtained, especially on cast metals. I hope at some time later to be able to give more detailed information.

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[1]Director, Battelle Memorial Institute, Columbus, Ohio.

[2]Consulting metallurgical engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh.

[3]Engineer, Aluminum Company of America, New Kensington, Pa.



Showing Relative Size of 2¼-Inch and 0.05-Inch Diameter Specimens.

Author's Closure

Mr. Templin's comments on size effect in cast aluminum are interesting. It is hoped that he will carry this work further. Some tests made by Schwinning[4] on aluminum, bronze, and copper wires, varying from 1.75 to 4.55 mm. in diameter, showed an increase of endurance limit with decrease in size. This corresponds to tensile tests and is to be attributed to cold work in drawing the wire. In studying size effect, one must be careful to eliminate all other variables, such as cold work, surface finish, etc.

Professor Moore's comments on testing small strips are of interest. The Bell Telephone Laboratories have done a great deal of work in the testing of thin strips.[5] We have made some use of a strip fatigue testing machine[6] at the Westinghouse Research Laboratories. The machine operates at constant deflection but is not suitable for general use, because of the change of the stress-strain curve with stressing for certain materials.

Quite recently, a rather interesting application arose for the use of the small machine. The question of fatigue at low temperature was raised in connection with traction apparatus operating in cold climates. Tests were run at 10 degrees Fahr. by placing the entire machine in a refrigerator coil. No bearing trouble was encountered and indications are that the machine could have been operated at considerably lower temperatures.

Someone raised the question of cost of specimens. Absolute cost has very little meaning because of dependence on the shop system, the accounting system, and the rate. Relative costs are of interest, however. Our first small specimens cost about 75 per cent of the cost of standard McAdam type cantilever specimens. It is the writer's opinion that this relative cost should not exceed 50 per cent.

[4]Zeitschrift für Metallkunde, October, 1929, p. 347.

[5]Townsend and Greenall "Fatigue Tests of Non-Ferrous Sheet Metals," Proc. A. S. T. M., Vol. 27, Pt. II, p. 153 (1927).

[6]See discussion of above reference.

CONCENTRATED PRESSURE AND SOME OF ITS APPLICATIONS TO THE ROLLING PROCESS

By Dr. A. Nadai

Abstract

In the following several cases of elastic distribution of stress produced by concentrated loads acting on the surface of bodies are briefly treated. The intention is, however, to try to apply some of the information obtained by analysis to cases of similar distributions of stress produced in the plastic state of ductile metals and to obtain by this more information about the processes taking place in the severely pressed parts of a rolled metal sheet during the operation.

Perfect Elasticity and Plasticity

TO define elasticity and measure it as a mechanical property of matter, it was advantageous and necessary to first test the materials under simple homogeneous states of stress, such as pure tension, pure compression or pure shear in order to show how stress depends on strain. By means of the elementary tests, it was early found, that if the stresses do not exceed certain limits and if the changes in shape remain small compared to the dimensions of the bodies, the deformations for many materials of construction are proportional to the forces producing them. It was also found that only two independent material constants determine the property of elasticity in the isotropic materials, in which the elastic properties do not depend on the direction of the applied stress. In other more complex states of strain or stress, produced by more complicated systems of external forces or in bodies of changing dimensions, a similar thing was found. The more complicated cases could be described, because from the simple cases of stress a general law of elasticity could be derived, by means of which the states of strain could be expressed in terms of the states of stress. It is perhaps sometimes not quite recognized that the

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present theory of elasticity as used by engineers as well as by physicists or mathematicians is based on a few idealizations of some properties of matter, which in truth can never follow the laws attributed to them exactly on account of all the imperfections encountered in the actual materials of construction, not to mention the cases, in which the laws are only a first approximation. For such a material as soft steel, we know that the strains remain "purely elastic" within the limits of errors and the six components of strain can be expressed as linear functions of the six components of stress, provided that the stresses do not reach the limit of plasticity or the yield point.

Now, an idealized state of equilibrium in the plastic state or "a perfectly plastic mass" may be considered by assuming some few characteristic or essential properties attributed to it just as are assumed, in "the perfectly elastic isotropic material." It is known that certain ductile metals have the remarkable property of exhibiting a well defined yield point. This means that if the stresses do not reach certain values, the strains remain perfectly elastic and if the yield point is reached, plastic or permanent deformations start to develop. The idealization introduced shall be to assume that while plastic flow occurs, the stresses do not change and remain independent of deformation as well as of velocity of deformation. Tests have shown, that within some few per cent of unit extension or unit shear, plastic flow under low temperatures in a ductile metal such as soft steel, may be considered as approaching these idealized conditions, if great velocities of deformation, such as may be produced by impact in connection with plastic flow or during quick rolling or drawing of a specimen are at first excluded.

For a perfectly plastic mass, it is possible to express rules regarding the stress and strain, which in some aspects are quite analogous to the well known law of elasticity as contained in the stress-strain relations of a perfectly elastic material. It should be emphasized here, that the great simplicity, which might be attained by using certain assumptions regarding plastic flow justifies their introduction perhaps even in such cases where departure from the idealized conditions is more apparent.

In the case of a slow stationary plastic flow there exists a relation containing only the components of stress, which is satisfied. One further idealization which might be introduced in our

definition of a perfectly plastic mass is that plasticity shall not depend on the average pressure to which an element of a plastic body is subjected, that is, if the normal stresses acting on an element are all increased or diminished by the same pressure, this shall not affect the reaching of the limit of plasticity. Designating by s_1, s_2, and s_3 the principal stresses and by s_0 the yield stress in pure tension from the property just mentioned and attributed to a plastic mass, it immediately follows that the relation by which the reaching of the limit of plasticity is expressed can only contain the differences of the principal stresses s_1, s_2 or s_3. The condition of plasticity

$$(s_1 - s_2)^2 + (s_2 - s_3)^2 + (s_3 - s_1)^2 = 2s_0^2 = \text{constant}$$

seems to express many results of tests in a satisfactory manner. To this condition five other equations may be added, which express the stress-strain relations valid for a plastic mass and take the place of the six linear relations contained in Hooke's generalized law of deformation of a perfectly elastic material. We will not write down these relations here, as they can be found elsewhere, but only point to one fact, namely that while for an elastic material there exists a single valued correspondence between the states of stress and of strain, this is no more the case for a perfectly plastic mass. There are only five equations available combining the six components of stress with the six components of strain and leaving thus one component of strain undetermined. By this the experimental fact is expressed that the deformations may change while the stresses remain unchanged. It is perhaps worth while to note that plasticity under these idealizing assumptions apparently depends only on a single material constant, namely the yield stress in pure tension s_0 (or some other equivalent stress for yielding under other states of stress), provided that the material can be considered as incompressible (in which case a second material constant contained in the stress-strain relations mentioned above, namely the ratio expressed by the lateral to the axial elongations is equal to $\frac{1}{2}$).

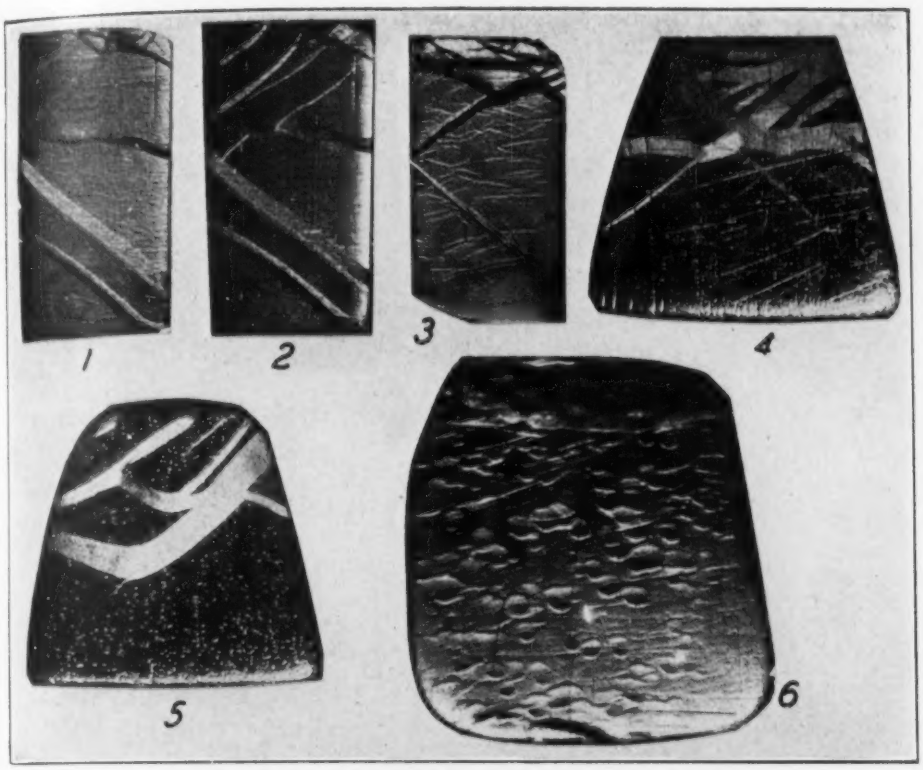
Concentrated Pressure

Among the various cases of distributions of stress, which have been worked out in detail on account of their practical applications in engineering either for an elastic material or in recent years also under the assumption of a perfectly plastic mass, a group of

cases deserves attention. These are the states of stress produced by loads concentrated on comparatively small portions of the surface of the stressed bodies, which deserve special attention because of the close relation of these cases to certain important mechanical properties of the materials of construction or to certain manufacturing processes. To the first belongs the question of a more satisfactory explanation of what is expressed by the word hard or the property hardness in solids and especially in the ductile metals. Under the latter are to be considered the manufacturing processes, which utilize the concentration of pressure for the purpose of forming thin rods, wires or sheets from compact pieces by rolling or drawing. Among the great number of elastic stress distributions which have been analyzed more exactly, a considerable amount of work has also been spent to find the distributions of stress produced by concentrated loads or pressures. Unfortunately in the earlier days, it was thought that by such an analysis the property of hardness could be defined on a more scientific basis. It was especially the great physicist H. Hertz[1] who about fifty years ago tried to work out a definition of the hardness on the basis of the theory of elasticity. It is known since these early attempts that it was not possible to define hardness satisfactorily, as Hertz thought.

If two perfectly elastic bodies with convex surfaces are brought in contact and pressed against each other by two forces in the direction of the common normal, the surfaces become somewhat flattened near the point of contact and as a result, stresses of considerable value are set up. If we imagine according to Hertz the convex surfaces in the neighborhood of the point of contact replaced by two flat ellipsoids, the distance between opposite points of the surface at the instant of mathematical or point contact is a homogeneous quadratic function of the co-ordinates x and y of these points taken with respect to a right angled co-ordinate system in the plane of contact. Hertz determined the shape of the surface of contact and the distribution of stresses in this surface and its neighborhood. He found that for two perfectly elastic materials, the area of contact was an ellipse and that the distribution of the pressure in the compressed surfaces was given by the ordinates of half of an ellipsoid above one of its principal planes. On the basis of his calculations, Hertz proposed to define as the measure of hard-

[1]Gesammelte Werke, Vol. 1.



Figs. 1 to 6—Lüders Lines and Phenomena of Slip in Mild Steel. Photographed on the Polished Surface by the Schlieren Method.

ness of the material the value of compressive stress at the elastic limit in the middle of the area of contact of two equal balls compressed together. On account of the difficulty of determining the elastic limit at such a hidden point, as the center of an area of contact, on account of the cracks arising at the edges of the compressed areas in case brittle materials are tested, when two balls are pressed together, as well as in the case of ductile materials on account of the almost unavoidable plastic compression, his method could never be used.

It is known that under the impression of a hard body, for example, a hard steel ball under the smallest pressures in materials such as the ductile metals, plastic deformations begin to develop and the impression or the indentation observed is rather the result of a plastic than an elastic phenomenon. This is even the fact for brittle materials, for example, cast iron, which has a certain strength and will show permanent plastic deformation under concentrated pressure. It is in this fact, namely the simultaneous occurrence of

elastic as well as permanent plastic deformations or strains and both of about the same order of magnitude produced by the penetrating action of a rigid punch there lies perhaps the greatest difficulty, which has prevented a further application of the methods of stress analysis to these cases and has impeded further progress in analyzing the distributions of stress produced by concentrated pressure so important to many branches of engineering[2].

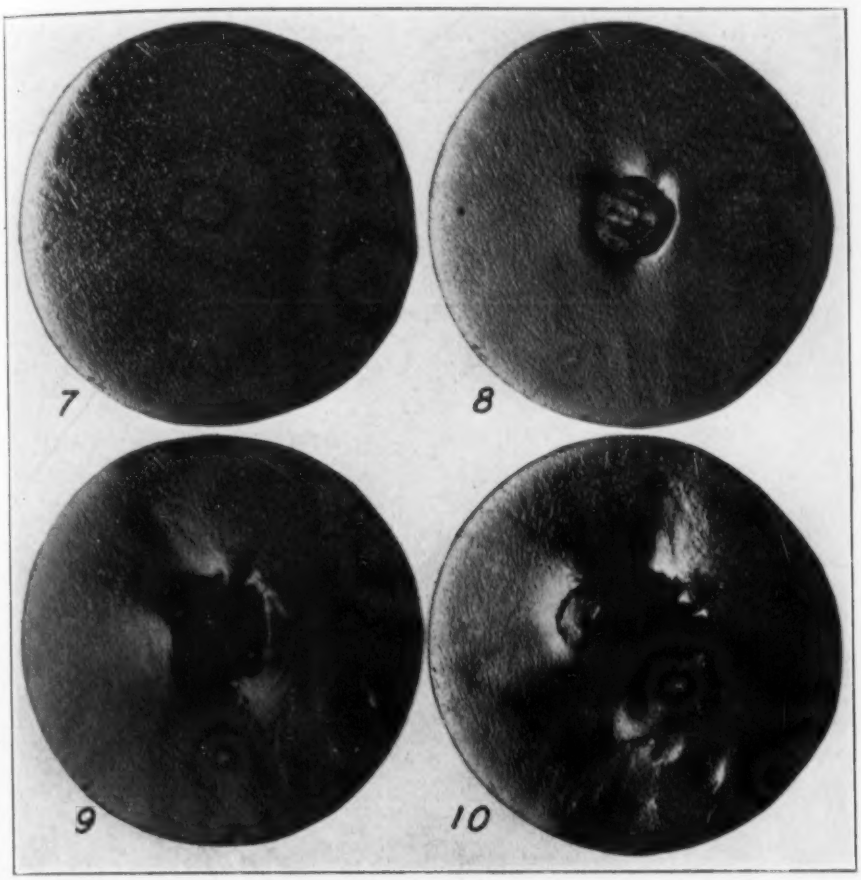
Formation of Surfaces of Slip in Plastic Bodies Under Plastic Impressions

The circumstances mentioned above suggest a search for methods, by means of which the nature of these distributions of stress might be studied by direct testing.

Some valuable information may already be obtained by direct visual observation of the slight distortions on the surface of test specimens produced by penetration of fairly rigid punches in soft metals or other soft materials. A steel punch with a hardened cylindrical end was forced into test pieces of cast zinc, soft and hard copper, and soft iron. As will be seen from the following photographs, the various metals behave differently when an indentation is made by a punch. To observe the distorted surface, the test pieces were polished before the test, then the impression made and photographed. To obtain photographs of the relief on the highly polished and therefore reflecting metal surfaces, the author used a method quite similar to the so-called "Schlierenmethod," by means of which small changes in the density of a gas or air in the sound waves can be made visible, or the movement of air caused by a flame[3]. The application of this method is illustrated by some photographs of compression test pieces of mild steel in Figs. 1 to 14. The test specimens were polished on their surfaces and compressed until the first signs of a plastic deformation became visible on the mirror-like polished faces. The photographs 1-6 of these surfaces show the fine relief caused by the strips of slip (Lüders lines). The intersections of the thin layers of plastically deformed steel with the polished surface can be seen in Fig. 1 to 6 as well as

[2]The great amount of valuable information regarding the methods how to test hardness, which have been gathered since the time of H. Hertz and a critical review of these methods and of other correlated work can not be mentioned here. These subjects lie beyond the scope of this paper. In this connection, a noteworthy paper of L. B. Tuckerman shall be referred here ("Hardness and Hardness Testing", Mech. Eng., Vol. 47, 1925), in which several facts regarding a mechanical definition of hardness were clearly brought out.

[3]Schweizerische Bauzeitung, Zurich, Vol. 83, Nos. 14 and 15, 1924.

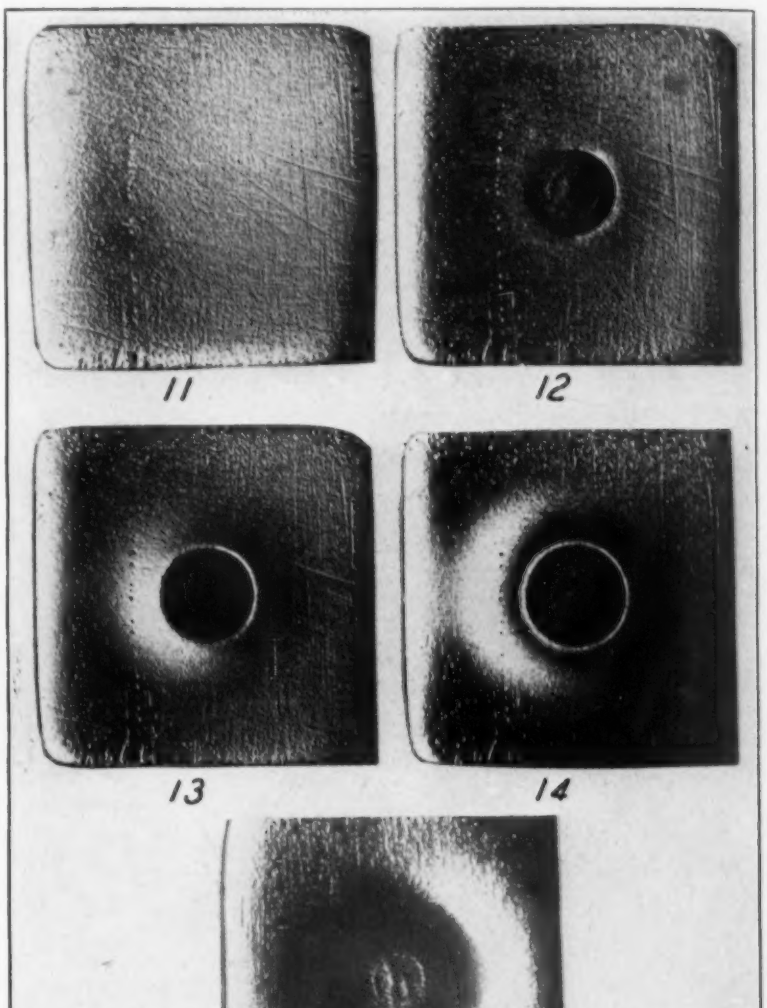


Figs. 7 to 10—Photographs of Impressions of a Cylindrical Punch in Cast Zinc.

the smallest irregularities (scratches, minute grooves) on the polished surface, which are reproduced exaggerated in the photographs.

Impressions of a Hard Cylindrical Punch

Zinc: The surface of the undeformed test piece is shown in the upper left figure of Figs. 7 to 10. On account of the low tensile strength of zinc cracks are soon formed in a radial direction which can scarcely be recognized in the photographs. By these cracks the distortion of the surface is disturbed and does not develop symmetrically around the axis of the test piece. It is perhaps noteworthy that the grooves and markings produced by the lathe tool reappear again in some parts of the more severely distorted surface around the indentation of the punch, although the surface has been finely polished. The reappearance of these grooves is caused by the



Figs. 11 to 14—Impressions of a Cylindrical Punch in Hard Copper. Fig. 12—67,000 Pounds per Square Inch. Fig. 13—90,000 Pounds per Square Inch. Fig. 14—111,100 Pounds per Square Inch. Fig. 15—Impression in Soft Copper.

work hardening of the zinc along the lines drawn by the tool. Under the new plastic deformation caused by the compression of the punch, the work hardened circular grooves are a little less deformed than the soft portions and project therefore through the well polished surface. After the compressive stress was increased to about 67,000 pounds per square inch, the surface became badly dis-

torted, since the cast zinc on account of its small tensile strength could not withstand the tensile stresses set up in the tangential direction, fine radial cracks opened.

Copper: By the impression of a cylindrical punch in hard copper, a bulging around the indentation is caused, which may be noted in the photographs taken after severe compressions in Fig. 12 to 14. First, in the left upper corner again, the surface of the unstressed piece is shown. The scratches on it as well as the other details appear in these photographs greatly exaggerated on account of the sensitive method; the surfaces were in truth well polished and reflected as a mirror. Under 111,000 pounds per square inch severe bulging is visible. In Fig. 15 for the sake of comparison, the indentation of the same punch is shown in a very soft piece of copper. This material was forced downward and a funnel shaped hollow appeared.

Mild Steel: In the neighborhood of the impression a very regular system of lines may often be seen. These lines cross the radii approximately at a constant angle of about 45 degrees and form therefore a system of nearly orthogonal logarithmic spirals. In the surface layer, the directions of the principal stresses are in the radial and tangential directions; the directions of the maximal shearing stresses cross the radii therefore at 45 degrees and coincide with the logarithmic spirals visible in the photographs. These slip lines are characteristic for steel and may often be observed in remarkably regular form as in the two examples shown in Figs. 16, 17 and 18.

Paraffine: This material has the valuable property of showing the slip lines well if deformed permanently. The paraffine used in the compression tests was dark. Where it was strongly deformed beyond the plastic limit or where layers of slip were formed, the paraffine appears discolored and almost white.

In Fig. 19 the impression produced by the penetration of a steel cylinder into the soft paraffine can be seen. As this material has practically no tensile strength, again the radial cracks appear around the impression, where tensile stresses must have acted in the tangential direction. It is perhaps noteworthy that apparently these radial cracks do not penetrate to greater depth; they are more of a surface phenomenon. This can be seen in photograph Fig. 20, which shows the longitudinal section of a paraffine cylinder

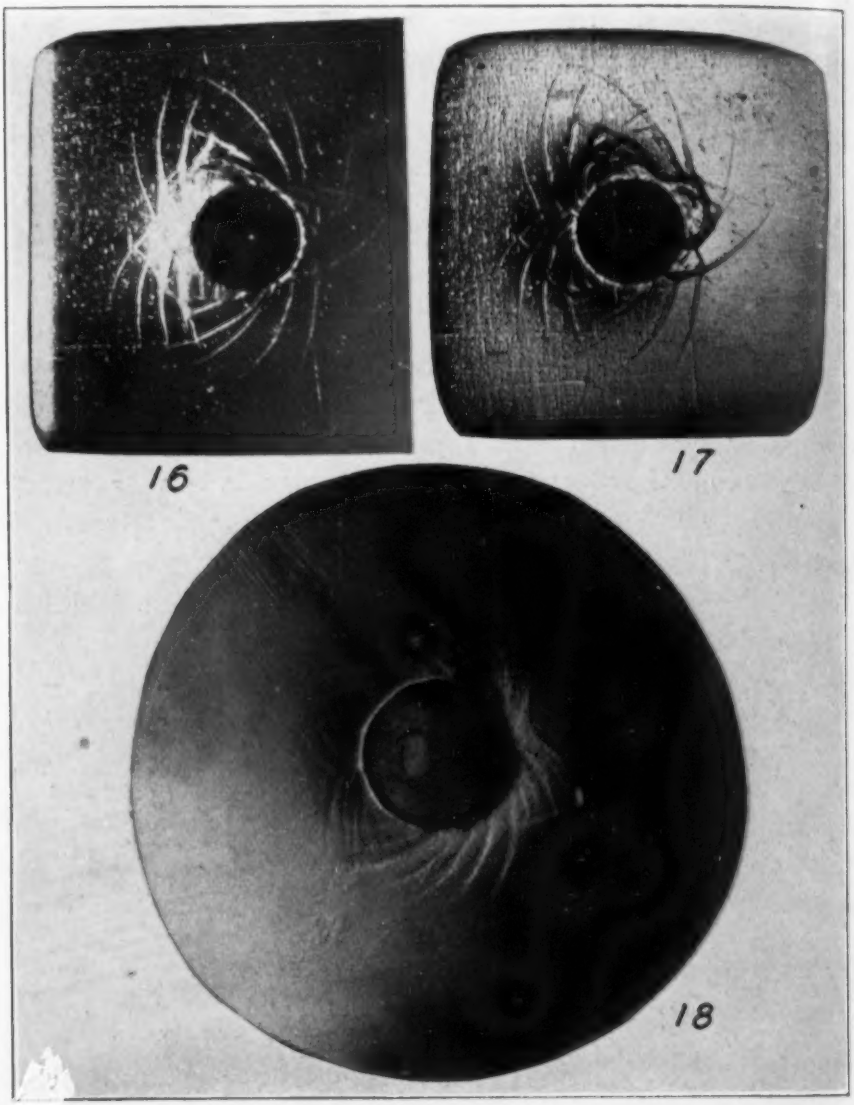


Fig. 16 and 17—Impression of a Cylindrical Punch in Soft Steel. Photographs are of Same Impression with Different Illumination.
Fig. 18—Photograph Showing Regular System of Slip Lines Around the Impression.

after an impression. In the section, a white crescent appears, pointing with its peaks towards the corners of the punch. Along this white region the material was stressed the most severely.

In Figs. 21 to 24 can be seen the structural changes produced on the surface and inside of a truncated cone compressed under a rigid plate in the axial direction. On the surface (Fig. 21) a very regular system of slip lines appears consisting of a system of nearly orthogonal helices. This shows clearly that in such a compression

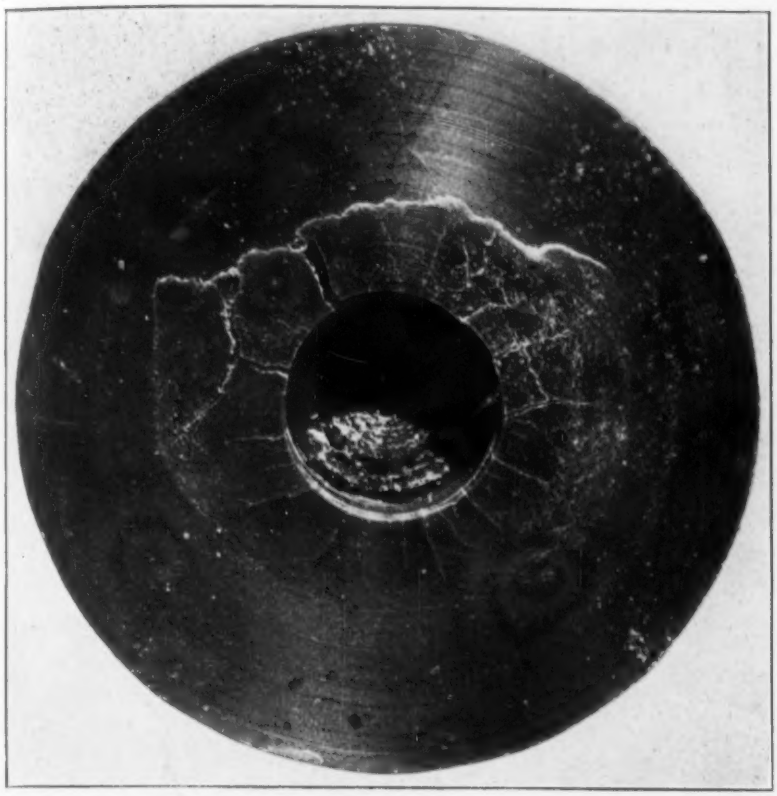


Fig. 19—Photograph of Indentation in Paraffine Showing Many Radial Cracks.

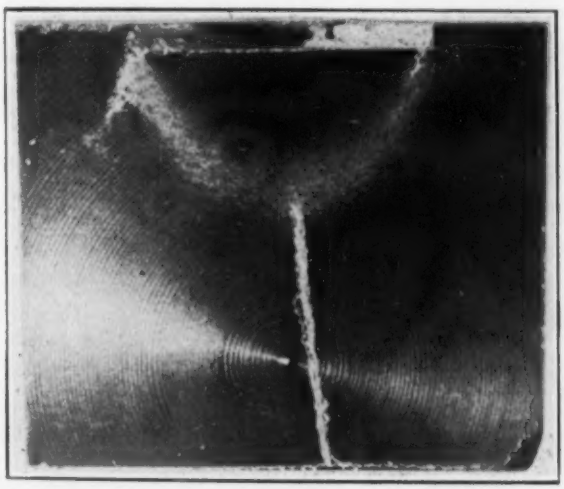
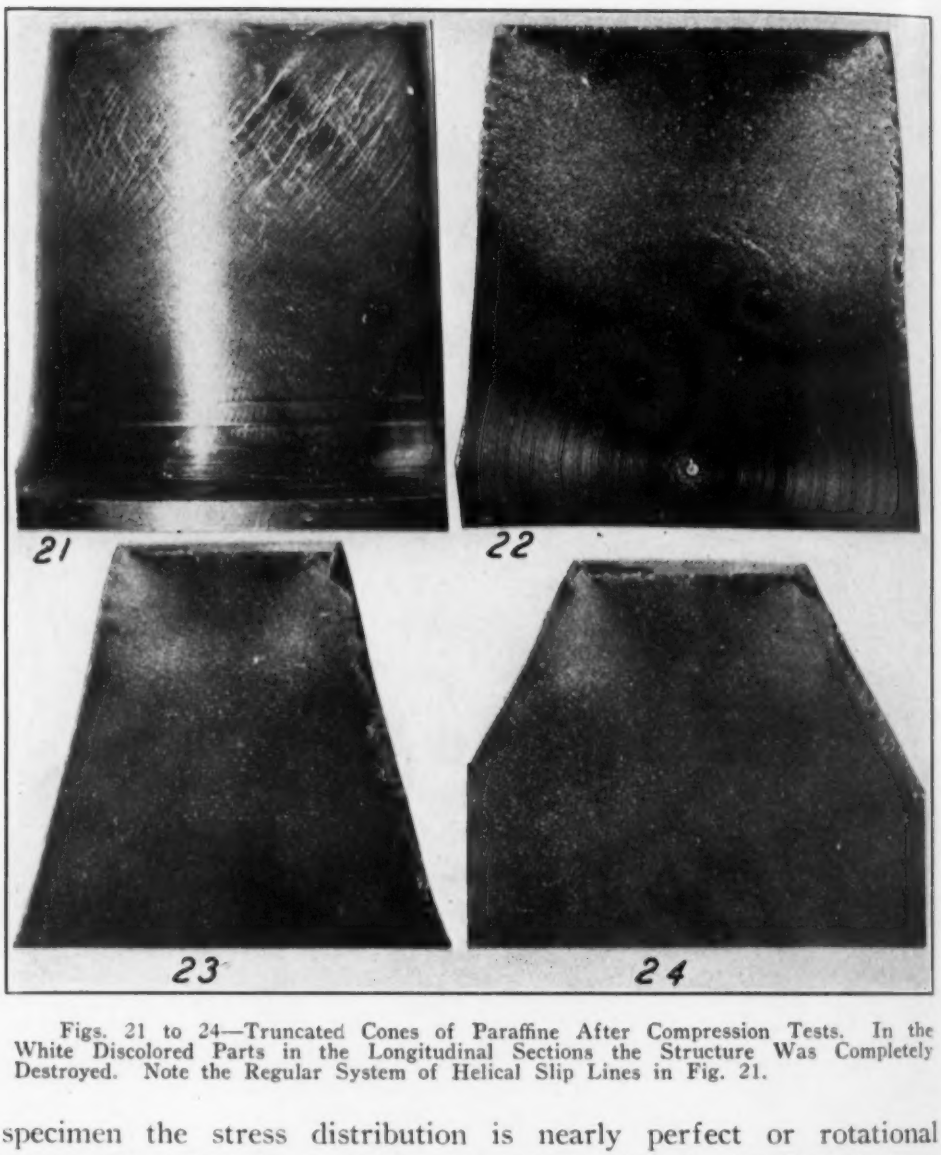


Fig. 20—Longitudinal Section Through a Cylinder of Paraffine. The White Arc Under the Impression Was Severely Strained.



Figs. 21 to 24—Truncated Cones of Paraffine After Compression Tests. In the White Discolored Parts in the Longitudinal Sections the Structure Was Completely Destroyed. Note the Regular System of Helical Slip Lines in Fig. 21.

specimen the stress distribution is nearly perfect or rotational symmetry. That the stresses can not be uniformly distributed along the circular cross-sections of the cylinder is demonstrated by the longitudinal section of the same test piece shown in Fig. 22, in which the two white sectors pointing against the edges of the cylinder were much more severely stressed than other parts of it. This effect is caused by the friction acting between the rigid compression plate and the plastic mass below it, which tends to prevent the radial expansion of the soft cylinder thus causing a nonuniform distribution of stress in it.

These remarks may be generalized at once to the statement that in all compression tests, in which a fairly rigid plate is compressed against a soft material, the friction in the surface of contact prevents the lateral expansion of the material below it thus causing a nonuniformity in the distribution of stress below the surface of contact. Similar effects to that in Fig. 22 can be seen in Figs. 23 and 24.

Strain or Flow Figures in Mild Steel

The examples mentioned above showed that in plastic materials under certain conditions very regular figures can be observed which apparently have a close relation to the states of stress which must be considered as their causes. In soft paraffine, these lines could easily be observed. Among the ductile metals, mild steel exhibits similar phenomena. The fine dull lines which can be observed on the polished surface of a mild steel specimen at the instant of the first yield or just a little later and which are known to engineers as flow or strain figures or as "Lüders Lines" give valuable information on the phenomena taking place in the structure of steel at the instant it yields[4].

One property of the flow or slip layers has an especial bearing on the mechanics of the plastic state. The surfaces of the thin slip layers, in which the iron is apparently more severely deformed than in the adjacent material, coincide approximately with two of the surfaces of principal shearing stress (under these latter the maximum shearing stresses are meant, which correspond to a state of stress given by its three principal stresses s_1, s_2, s_3). In a tensile test, for example, they form an angle with the direction of tension (the principal stress which is not zero) which is a little greater than 45 degrees, (about 47 degrees) and in the case of compression usually a little less than 45 degrees (about 43 degrees). Neglecting these small differences, we may say that the slip layers make an angle of 45 degrees with the tension or compression in these tests. Since these properties of the flow layers also are exhibited in complicated three or two dimensional distributions of stress (that is, in the

[4]The formation of the flow figures in steel, described in older books on strength of materials and among others especially by Martens, Heyn, O. Mohr, C. Bach, and L. Hartmann has recently been studied again. Among others those who have studied them recently T. H. Turner and T. D. Jevons (*Journal*, Iron and Steel Institute, No. 1, Vol. III, p. 169), E. W. Fell (same journal, 1927), Pfeil (*Engineer*, London), Seigle (*Revue de Métallurgie*, Paris), might be mentioned here.

cases where the state of stress depends on all three, or at least on two of the co-ordinates x, y, z, of a point of the stressed body), observations relative to their position form a valuable aid in the investigation of states of stress in plastically deformed solids. For this reason they will be used in that which follows. For observing the flow figures either the method of A. Fry of etching steel or that mentioned previously of observing them on polished specimen in reflected light will be used.

An example of a stress distribution of rotational symmetry has been mentioned above, in the case of which the strain figures could be observed near the impression. (See Figs. 16, 17 and 18). After what has been said, it is obvious that in case of a rotational symmetry the stresses in the plane surface near the plastic impression can only depend on the distance r of a point from the axis of the compression, or the center of the circular indentation. Furthermore, in the plane in which the concentrated pressure has been applied they will consist of (1) a radial distribution of normal stresses designated by the letter s_r (radial stress), (2) of a distribution of normal stresses acting tangentially to any circle $r = \text{constant}$ and designated by the letter s_t (tangential stress), (3) of a distribution of axial normal stresses, acting parallel to the direction of the pressure, designated by s_z. The stresses s_z will be equal to the pressure p if r is smaller than the radius $r = a$ of the punch, and $s_z = 0$ if $r > a$. Besides these normal stresses $s_z = p$, in general a shearing stress will act s_s in a radial direction in the pressure area caused by the friction. On account of the radial symmetry, one sees at once that for $r > a$ in the plane of contact, but outside of the pressure area s_r and s_t are principal stresses, and hence one system of the principal shearing stresses will act at an angle of 45 degrees to the radii. The curves connecting elements inclined at an angle of 45 degrees to the radii are two orthogonal systems of logarithmic spirals as shown by Fig. 25. These logarithmic spirals were observed as slip lines in the photographs in Figs. 16, 17 and 18.

By means of the theory of elasticity, the stresses s_r, s_t, and s_z may be computed for the case of a concentrated pressure applied in a plane boundary of a piece of elastic material, and under the assumption that the pressure $s_z = -p = f(r)$ in the area of contact $r < a$ only depends on the co-ordinate r, or a function of r. They are given by the formulae:

$$\text{axial stress: } s_z = -p = f(r) \qquad \text{(for } r > a \text{ evidently } p = 0\text{)}$$

$$\text{radial stress: } s_r = -p + \frac{1 - 2\nu}{2} \cdot \overline{p}$$

$$\text{tangential stress: } s_t = -2\,\nu\,p - \frac{1 - 2\nu}{2} \cdot \overline{p}$$

where $\overline{p}$ designates the mean pressure within a circle having r as its radius, or

$$\overline{p} \cdot r^2 = 2 \int_0^r p(u)\, u\, du$$

and ν is Poisson's ratio. If the pressure $p = 0$ for $r > a$, for all points $r > a$ in the surface $\overline{p} = P : \pi r^2$, where P is the load concentrated in the circle $r = a$.

These formulae permit computation of the stresses in the plane of contact just before yielding starts in an elastic body, provided that only a normal pressure acts in this plane (and no friction is acting in it) and provided that the distribution of the pressure is $p = f(r)$ only depending on the distance r from a given point of the surface.

Plane Distribution of Stress Under Concentrated Pressure. Shearing Stress Lines (Isochromatics) and Shearing Stress Trajectories (Slip Lines).

In the following, a number of cases of elastic contact problems in which the stresses depend only on two co-ordinates will be briefly discussed. In some of these cases, analytical expressions for the stresses are available, which have been established by mathematicians and engineers. The intention is, however, to compare the results of these calculations with certain results obtained from photoelastic tests and also with the slip lines observed under similar loading conditions in mild steel after the yield point has been reached and the plastic deformation begins to develop.

On the author's suggestion, G. Mesmer[5] at the University of

[5]The photographs and figures (Figs. 27-49) showing the lines of slip were prepared by G. Mesmer and are published in his doctor's thesis of the University of Goettingen,—Zeitschrift für Technische Mechanik und Thermodynamik, Vol. 1, Nos. 2 and 3, Berlin, 1930.

Goettingen has investigated the formation of slip lines under concentrated loads. It was G. Mesmer's suggestion to apply the photoelastic method for the purpose of comparing the results obtained on an elastic material with those of tests in which the yield point has been reached under similar loading conditions in mild steel.

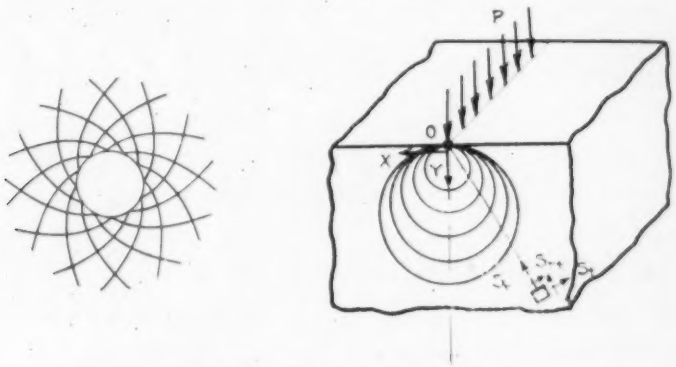


Fig. 25—Logarithmic Spirals as Slip Lines.
Fig. 26—Michell's Radial Stress Distribution. Single Force Distributed Along Straight Line. The Isochromatics are Circles Passing Through O and Tangent to the X Axis.

If a transparent specimen in the form of a thin sheet is stressed in its plane and linearly polarized light is sent through the stressed specimen by means of a nicol prism, interference phenomena or colored fringes can be produced and observed on a screen by the use of a second nicol. The credit for having developed the photoelastic method belongs principally to Prof. E. G. Coker of London, to whom reference will frequently be made in the following. In the United States among others especially R. V. Band has applied this method of studying stress problems. What is essential here is that all points of a transparent test piece, which are submitted to the same difference of principal stress, the same optic effect is produced on the screen, on which the illuminated test piece is projected. Hence on the screen certain colored lines will appear in regular repetition and a constant stress difference will correspond to each color. On an ordinary photographic copy of the colored picture black and white lines will appear, the black and the white lines corresponding to certain colors of the colored fringes or certain given differences of principal stress and these differences may be determined by suitable calibrating methods.

In a thin sheet stressed by forces in its plane the normal stress which is perpendicular to the plane of the sheet and the shearing stresses acting in this plane are zero and hence this direction is a

principal stress direction. We call isochromatic lines, the lines connecting, in an image of the test piece, points having the same color on the screen and corresponding to curves along which the difference of the two other principal stresses s_1 and s_2 is a constant:

$$s_1 - s_2 = \text{constant}$$

These curves are also lines of constant principal shearing stress, because one of the three principal shearing stresses is equal to one half of the above difference. The isochromatic lines may also be called shearing stress lines.

From the isochromatics or lines of constant principal shearing stress, we must sharply distinguish the trajectories of principal shearing stress called hereafter "shearing stress trajectories" or "slip lines." These latter have as their tangents the directions along which the principal shearing stresses act. They intersect the trajectories of principal normal stress at an angle of 45 degrees. The trajectories of principal shearing stress are two orthogonal systems of curves and they coincide approximately with the lines of slip along which the Lüder's lines appear in a mild steel test piece when the limit of plasticity is reached.

Several interesting two-dimensional cases having important practical application may be mentioned here. An infinite body extending indefinitely on the side $y > O$ of the x, z plane will be assumed.

Single Force Distributed Along a Straight Line and Acting on an Infinite Plane

Michell has worked out a solution for this case, which is remarkable not only on account of its extreme simpleness, but also on account of the practical applications of this case to more complicated cases of plane strain produced by concentrated loads. Using polar co-ordinates r and ϕ, the components of stress can be expressed as a radial normal stress s_r, a tangential normal stress s_t and a shearing stress s_{rt} (see Fig. 26). Michell found that the distribution of stress leading to a single force, which is distributed along a straight line and acting on an infinite plane (see Fig. 26) consists only of radial stresses:

$$s_r = -\frac{2P}{\pi} \cdot \frac{\sin \phi}{r}$$

The stresses s_t and s_{rt} are zero. In this formula P designates the force per unit of length acting on the surface.

For an experimental verification of this distribution, it is convenient to construct the lines of constant maximum shearing stress for this case. Along such a line the maximum shearing stress,

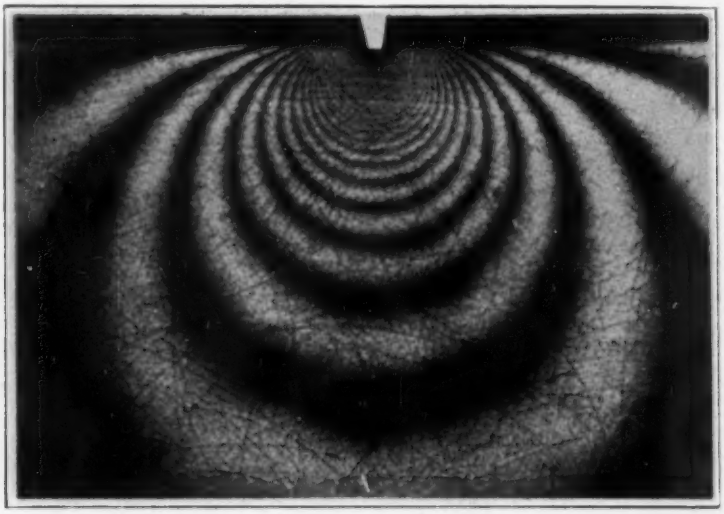


Fig. 27—Sharp Edge Pressed Against Plane. Michell's Radial Stress Distribution. The Isochromatics are Circles.

which is half of the principal stress difference or $(s_r - s_t) : 2$ must be constant:

$$s_r - s_t = -\frac{2P}{\pi} \cdot \frac{\sin\phi}{r} = c = \text{constant}$$

Taking the constant c equal to $c = -\frac{P}{2\pi a}$ from the preceding $\frac{\sin\phi}{r} = \frac{1}{2a}$

is obtained, which shows that the isochromatics or the lines of constant maximum shearing stress in this case are circles passing through the origin O or the point of application of the force P and having the boundary line y = O to their common tangent (compare Fig. 26). This is confirmed by the photograph Fig. 27 in which the black and white strips correspond to curves of the same color or to the isochromatics of a photoelastic test obtained with a celluloid sheet on which a single force was applied on one edge[6].

[6]Under Prof. Coker's numerous photoelastic tests, most of which are published in *Engineering*, London, (a short résumé of his tests may be found in the *Proceedings* of the International Congress for Applied Mechanics, Delft, 1924), the case mentioned above has also been tested.

Uniform Pressure Along Half Plane. (Fig. 28) In this case the principal stress trajectories form a system of orthogonal parabolas having the origin O as their common focus. The isochromatics or shearing stress lines are a pencil of rays passing through the origin O. From this last result we can learn that if a distribution of pressure

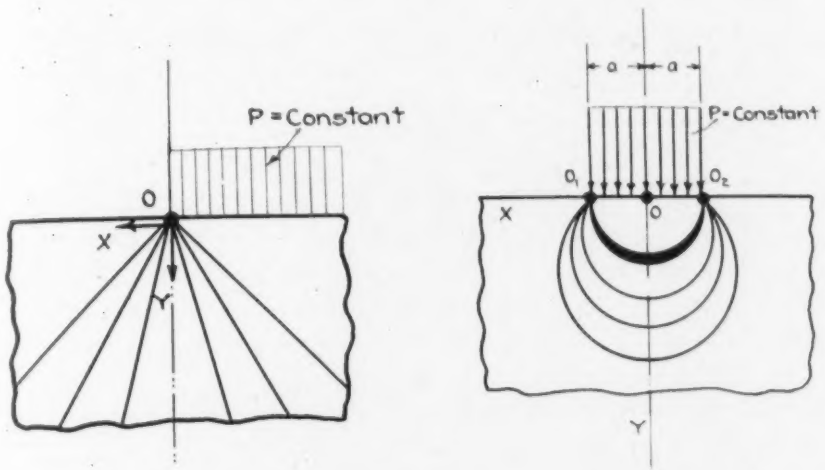


Fig. 28—Uniform Pressure Along Half Plane. The Isochromatics are a Pencil of Rays Passing Through O.

Fig. 29—Uniform Pressure Along a Parallel Strip on Infinite Body. The Isochromatics are Circles Passing Through the Points O_1 and O_2 and Having their Centers on the Y Axis.

acting on a plane surface of an elastic body tends to approach a finite pressure on the border of the surface of contact, all shearing stress lines will pass through the point which is the projection of this line in the Fig. 28. Such a point is a singular point of the stress distribution and in contact tests with ductile metals it has been found that the slip lines also pass through this point. The material at such a point is severely strained and cracks, or surfaces of rupture due to shear may often be seen starting from such a point. (Compare for example the cracks in the photograph of a compression test with paraffine in Fig. 20).

Uniform Pressure Along a Parallel Strip on Infinite Body. If a uniform pressure p on a plane boundary of an elastic body is distributed along a comparatively narrow strip, it can be shown that the isochromatic lines are again a system of circles. These circles pass through the points O_1 and O_2 (Fig. 29), and have their centers on the vertical symmetry line (the y axis). Along any of these circles the maximum shearing stress in the material has a constant

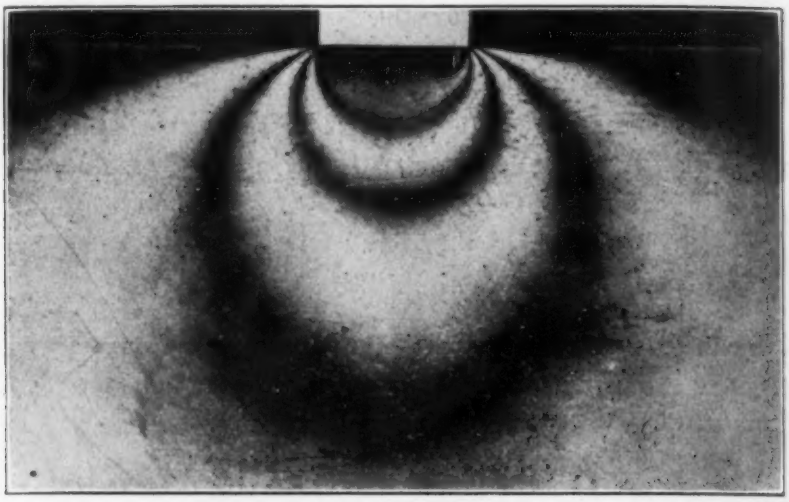


Fig. 30—Constant Pressure Under a Punch. The Isochromatics are Circles Passing Through the Two Corners of the Punch.

value. It is of interest to search for the circle on which the material will first yield. The greatest value of the shearing stress is found equal to

$$s_{max} = \frac{p}{\pi}$$

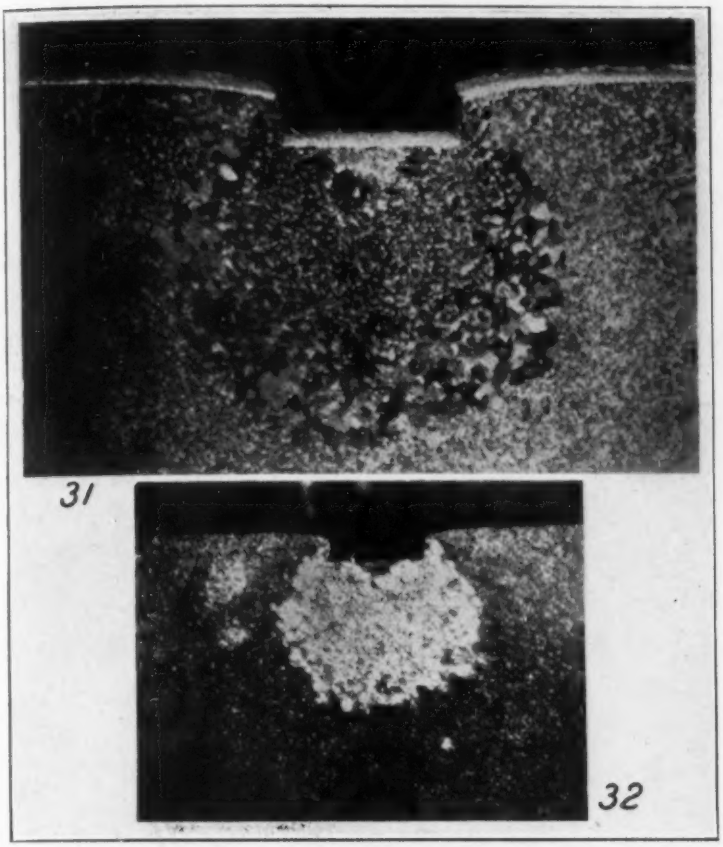
where p is the pressure per square inch in the contact surface, and acts on the semicircle having the middle point of the width O_1 and O_2 as its center. This circle has been shown in the Fig. 29 with a heavy line. On this circle the material will yield first under a pressure

$$p = \frac{\pi\, s_o}{\sqrt{3}} = 1.81\, s_o$$

where s_o is the yield stress for pure compression, provided that the pressure p is constant in the surface of contact and that no friction acts in it.

G. Mesmer was able to produce the isochromatics for this case by using certain precautions in preparing a special stamp of celluloid with slightly rounded corners (see Fig. 30) which was pressed against the test piece. The isochromatic lines thus obtained experimentally were found to be very nearly circles, passing through the corners of the punch as predicted by the theory.

It is perhaps of interest to note here that by means of other well-known changes in structure due to local plastic deformations



Figs. 31 and 32—Recrystallized Region in Section of a Steel Piece Under Impression of a Prismatic Punch. The Recrystallized Region Appears White in the Lower Photograph of a Fry Etching.

combined with a subsequent heat treatment similar observations may be demonstrated on metal pieces. This can be done by reheating a test specimen which has been previously deformed plastically until the grains recrystallize. Two such tests are shown in Figs. 31 and 32. These figures reveal the remarkable fact that the boundary of the recrystallized zone under an indentation and the lines connecting recrystallized grains of the same magnitude have a shape quite similar to the isochromatic lines of the photoelastic tests. The degree of exactness of this correspondence has not, however, been demonstrated as yet.

Rigid Punch. Using a fairly rigid metal punch having a perfectly straight edge on a piece of celluloid, a distribution of isochromatic lines is obtained as shown in Fig. 33. This indicates clearly that in this case (1) the isochromatics are not circles, and (2) that

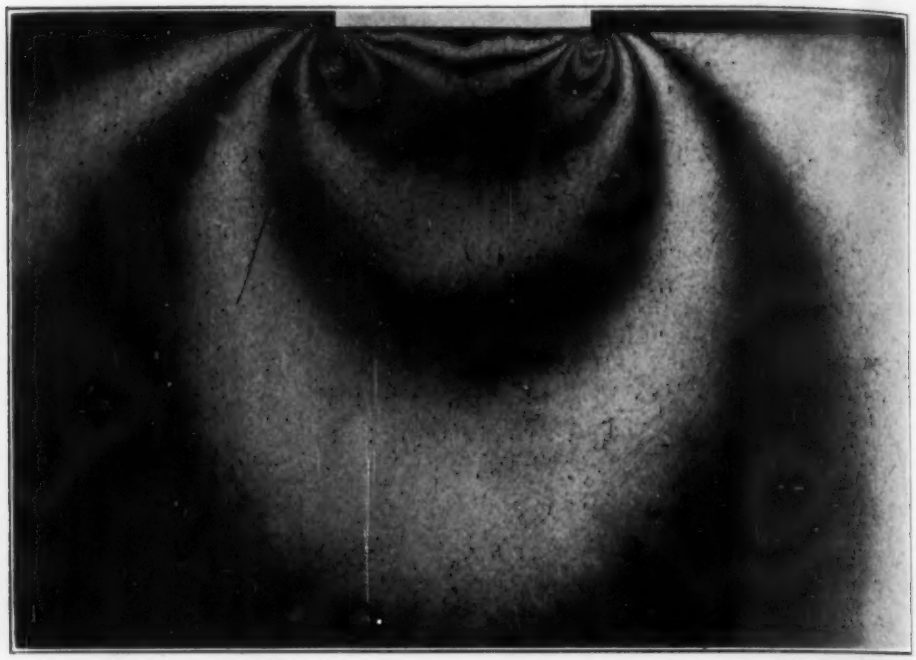


Fig. 33—Concentration of the Pressure and Stresses at the Corners of a Perfectly Rigid Punch, Shown by Photoelastic Test.

the stresses increase towards the two corners of the punch considerably. According to the elastic theory the pressure in the surface of contact must become theoretically infinite at the corners of the punch. Fig. 33 shows a great concentration of the stress by the repetition of the fringes at the corners. Hence immediately after the slightest application of a load under a perfectly rigid punch yielding must start at the corners of the punch. Consequently, a radiating system of slip or flow lines frequently can be observed in soft steel pieces under the action of concentrated loads and starting from the sharp edges of the punch. In more brittle material, fracture would start from these edges. Several observations of this kind will be quoted in the following.

Slip Lines. It is of interest to compare the shape of the trajectories of principal shearing stress in a perfectly elastic material with the shape of the slip lines as observed in similar cases of punching in mild steel. The former lines can be determined mathematically from the stress field if this is known, or experimentally if obtained through a photoelastic test, while the latter may be produced by a test on steel under concentrated pressure and be observed directly by suitable etching. The Figs. 34, 35, 36 and 37 show such

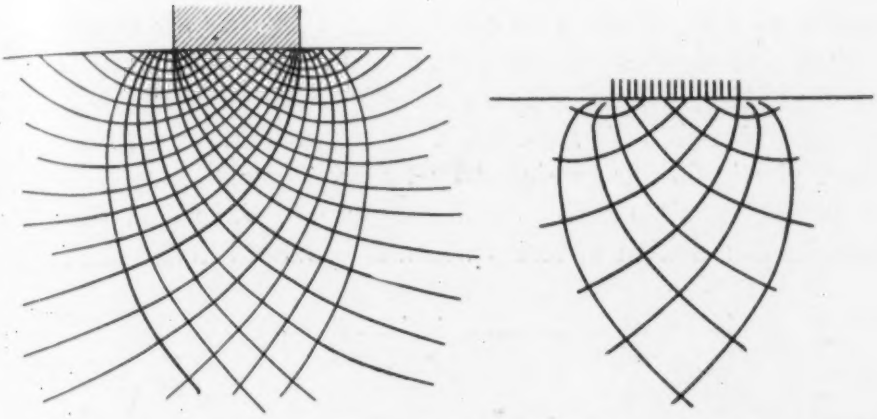
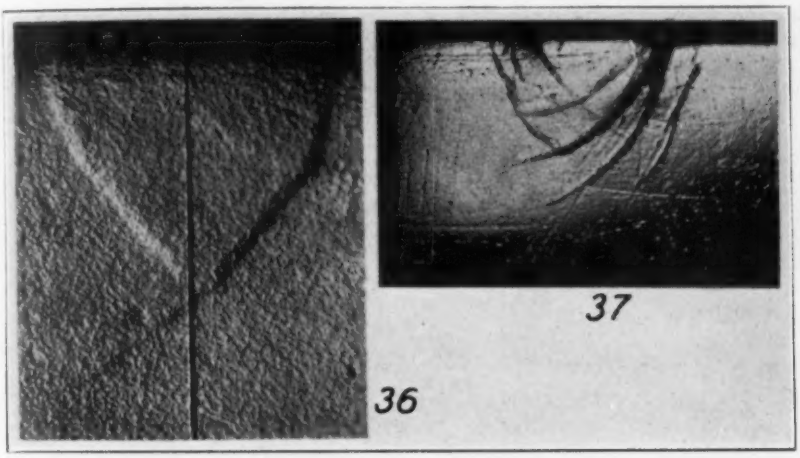


Fig. 34—Slip Lines for Concentrated Constant Pressure Determined Mathematically by G. Mesmer.

Fig. 35—Slip Lines Determined by Photoelastic Test for Constant Pressure in Narrow Strip.

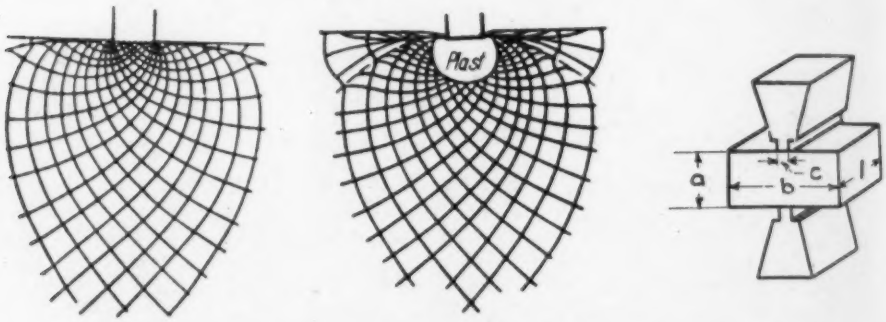


Figs. 36 and 37—Slip Lines Produced by Punch in Steel.

a comparison. In Fig. 34 the computed trajectories of principal shearing stress for the case treated above can be seen, and in Fig. 35 as obtained by a photoelastic test. In the etchings, Fig. 36, 37 the slip lines (which appear as dark lines or strips) can be compared. These figures and other observations by Mesmer indicate that as a first approximation the Lüders lines have a shape similar to the shearing stress trajectories in the most highly stressed portion under a concentrated load of an elastically deformed material under the same system of applied forces. How nearly exact this is, however, has not yet been thoroughly investigated. In Figs. 38 and 39 the slip lines can be seen obtained in photoelastic tests under the im-

pression of a rigid punch on celluloid. In Fig. 39 the pressure was so high, that the celluloid was yielding plastically under the punch.

Two Concentrated Loads, Plastic Flow in the Process of Rolling. If a thin sheet of metal is rolled between two rigid cylinders, plastic flow is produced by two concentrated loads acting in two parallel straight strips on the surface of the rolled sheet. To study the distribution of stress which is produced under this kind of



Figs. 38 and 39—Slip Lines Determined by Photoelastic Test Under Rigid Punch of Steel. Left: Under Small; Right: Under Comparatively High Local Pressure.
Fig. 40—Arrangement to Apply Two Concentrated Loads.

severe compression, and which is so much used in the process of rolling, test specimens of mild steel of the shape shown in Fig. 40, were severely stressed locally in compression by two hard steel punches. The method of yielding depends largely on the ratio of the width of the pressure surface to the thickness of the specimen measured in the direction parallel (and also perpendicular) to the direction of the compressive forces.

If the length l of the punch is several times (at least 5 times) the width c of the punch, assuming a uniform application of the load and close parallelism of the compression surfaces, there results in the middle part of the test piece plane plastic strain. (The stresses depend only on two co-ordinates). If, however, the length l is not very different from the width b a three dimensional strain distribution results. If l is small compared to c, the material flows away toward the free sides of the specimen and the plastic flow is entirely different from that for the case $l = \infty$ ("plane strain").

On the other hand, if the height "a" of the test specimen is comparable with the width c of the punch, the stresses produced by each of the two opposite punches influence those produced by the other. If, on the contrary, "a" is much larger than c, the stress

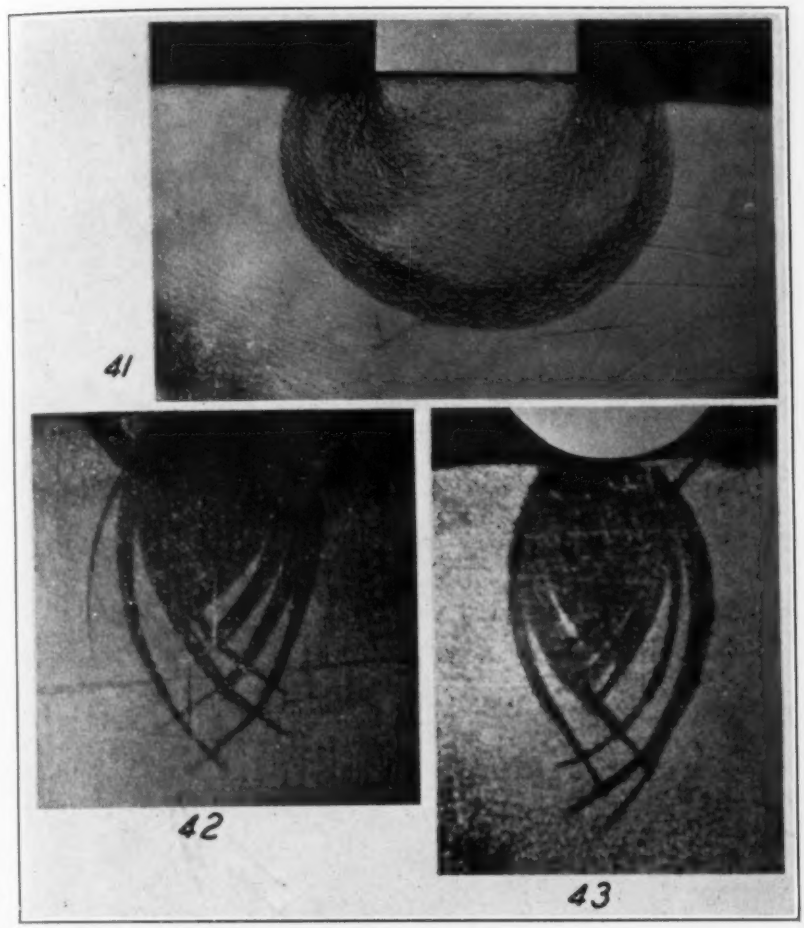


Fig. 41—Bulging of Surface of Thin Sheet Under Concentrated Pressure Along Narrow Edge.

Fig. 42—Slip Lines Under Impression of a Long Rigid Punch. (Etching.)

Fig. 43—Slip Lines in Steel Piece Obtained by Impression of a Hard Cylinder. (Etching.)

distribution in the neighborhood of the punch is about the same as if the second punch were not present and the first acted on an infinitely large test piece.

A few photographs of the regular markings observed on the sides of such test pieces of mild steel and of etchings of the plastically deformed portions in cross-sections are shown in Fig. 41. Such observations may perhaps help in analyzing the distribution of stress encountered in the process of rolling, for the manufacturing of steel.

The influence of the dimension l (measured parallel to the edge

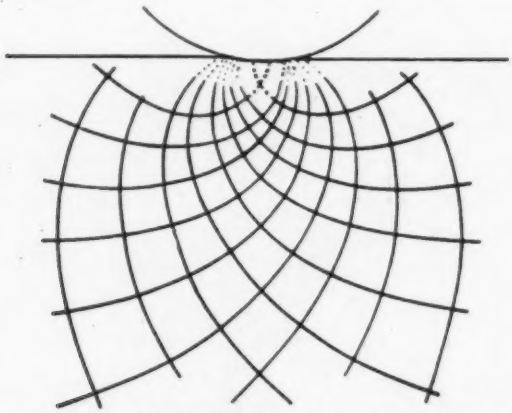
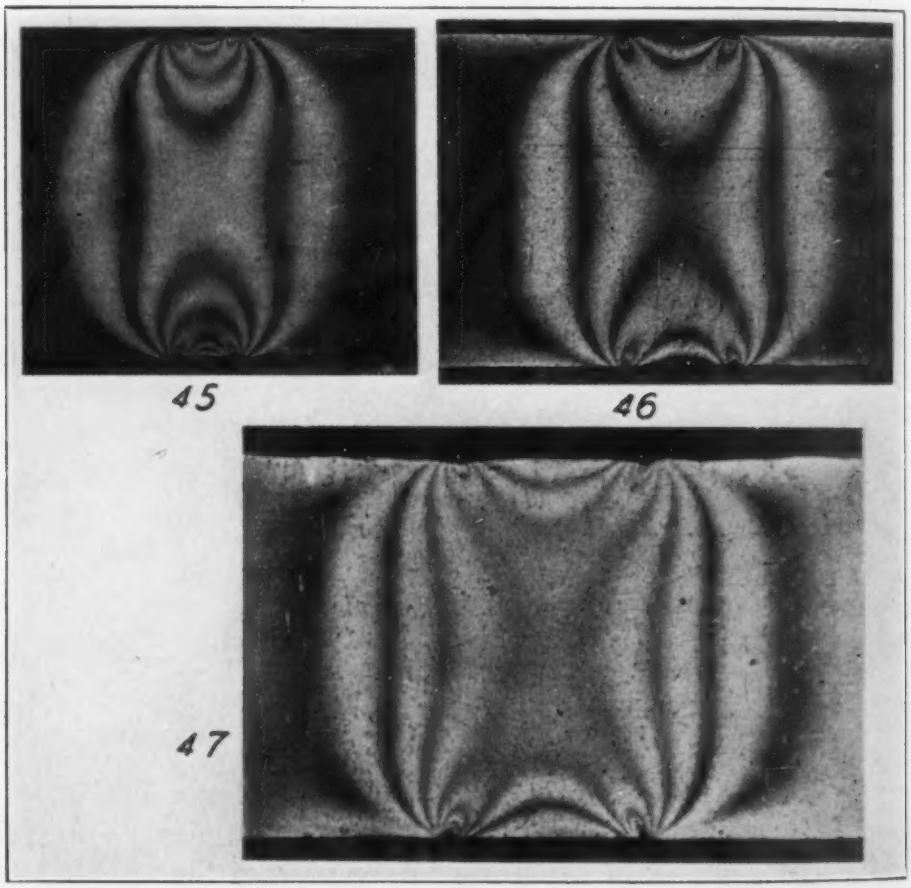


Fig. 44—Slip Lines Under the Impression of a Rigid Cylinder in Celluloid Obtained by Photoelastic Test.



Figs. 45, 46, and 47—Isochromatic Lines in Sheet Compressed Between Two Rigid Punches, for Ratios a:c = 3, 2, 1.
Fig. 45—a:c = 3, Fig. 46—a:c = 2, Fig. 47—a:c = 1.

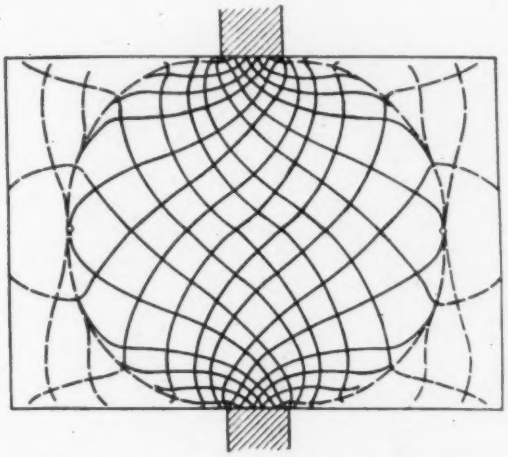


Fig. 48—Slip Lines Obtained by Photo-elastic Test.

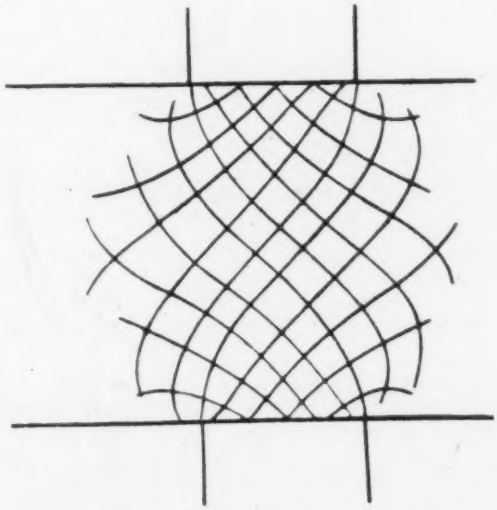


Fig. 49—Sliplines Obtained by Photo-elastic Test.

of the longest punch and perpendicularly to the plane of the photographs) is shown in Figs. 41 and 42. These represent essentially about the same case, namely, the action of a concentrated load distributed over a rectangular area of contact. If the load is applied along the narrow edge of a thin sheet, below the area of contact, both flat sides of the sheet will bulge out. The test specimen in Fig. 41 had a thickness $l = 1.1$ millimeters, a height $a = 37$ millimeters and a width $b = 45$ millimeters, the width of the punch was $c = 9$ millimeters. On both sides of the test piece slight bulges ap-

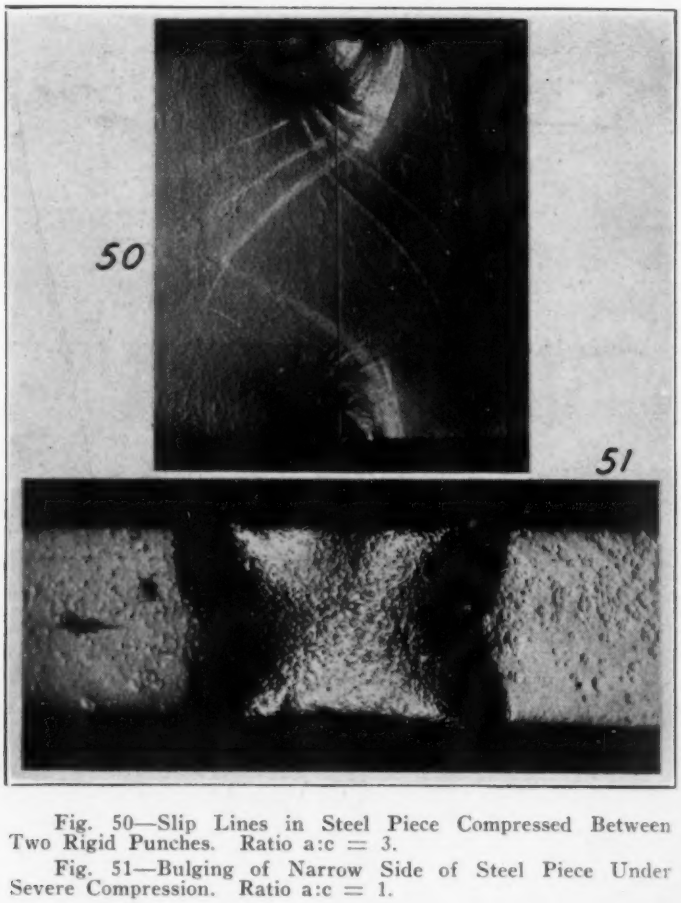


Fig. 50—Slip Lines in Steel Piece Compressed Between Two Rigid Punches. Ratio a:c = 3.

Fig. 51—Bulging of Narrow Side of Steel Piece Under Severe Compression. Ratio a:c = 1.

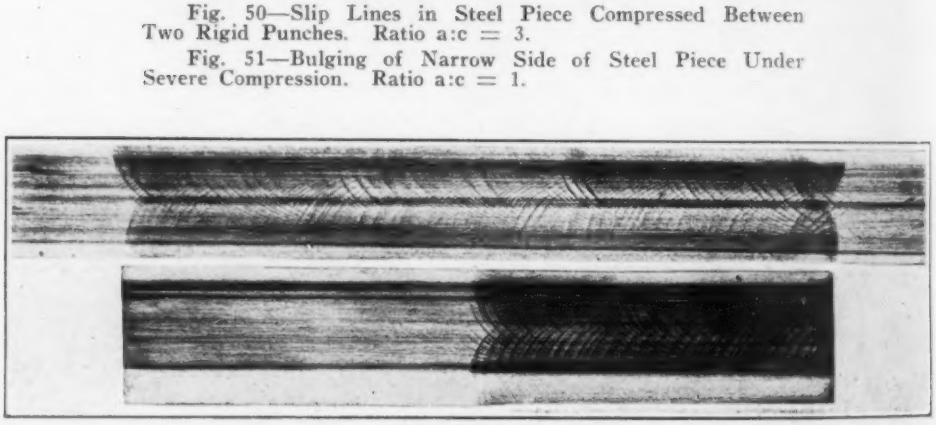


Fig. 52—Slip Lines in Longitudinal Section of a Cold Rolled Steel Sheet. According to H. Meyer and F. Nehl.

peared, such as the one shown in the photograph Fig. 41, which were bounded by curves similar to ellipses. The material tends in this case to flow away toward the free sides of the flat specimen. In the other case, where the length l was large compared with the width c of the punch, slip lines appear like those shown in the photo-

graph of an etched cross-section (Fig. 42) of the test piece. Now a wedge shaped plastic portion of the specimen is driven by the punch into the elastically reacting neighborhood and numerous slip lines appear. Another similar example can be seen in photograph (Fig. 44) of a Fry etching.

The impression of a long solid rigid cylinder on a soft steel piece is shown in section by the etching of Fig. 43. On the right in Fig. 44, are shown the shearing stress trajectories for the same case, as they were constructed from a photoelastic test by G. Mesmer with a celluloid test piece stressed purely elastically. The slip lines in the steel specimen apparently have a resemblance to the net of the trajectories as obtained from the photoelastic test.

The large influence of the ratio a/c on the stress distribution is clearly shown by comparing the three photographs of isochromatic lines in Figs. 45, 46 and 47 which were obtained for a ratio of the height "a" of the specimen to the width "c" of the punch equal a/c = 3, 2, and 1. The corresponding trajectories of shearing stress are shown in Figs. 48 and 49 for a/c = 3 and 2, and the lines of slip for the case a/c = 3 in Fig. 50.

A very interesting set of slip lines was obtained by H. Meyer and F. Nehl[7] in a cold rolled steel sheet, which can be seen in the photograph Fig. 52. These lines are gradually formed during the rolling of the sheet. When the rigid rolls travel along the surfaces of the sheet, they produce in the area of greatest compression a set of slip lines not greatly dissimilar to those in Figs. 50 or 48, and gradually, during the rolling, these lines proceed forward relative to the rolled sheet in a continuous manner.

Summary

In the preceding, the distribution of stresses in a number of cases of concentrated loads, have been studied by means of photoelastic tests in an elastic material and by a comparison of the shearing stress trajectories derived from such tests with the slip lines produced under similar loading conditions in mild steel under high concentrated pressure. It was thought that by such tests some information on the processes taking place in the severely compressed parts of a rolled metal sheet during the rolling operation could be obtained.

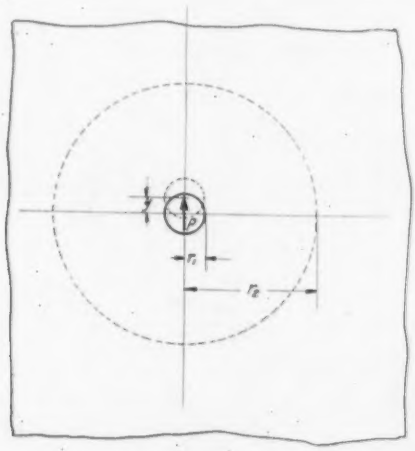
[7] *Stahl und Eisen*, 1925. Similar lines have been observed and described by E. Siebel in reports of the German Iron and Steel Institute at Dusseldorf, to which special reference must be made.

DISCUSSION

Written Discussion: By R. Soderberg, Power Engineering Department, Westinghouse Electric and Manufacturing Co., East Pittsburgh.

There is a considerable amount of information already available in elasticity and plasticity, which is certain to throw light on the difficult and obscure problems encountered in the process of rolling metals. It is only necessary that these are brought to light and re-arranged into the proper form. The present paper represents a very promising start of this work and the writer is looking forward to subsequent papers by the author on this subject with a great deal of interest.

I am particularly impressed by the usefulness of the Michell problem given on the seventeenth page. There are numerous cases where this solution is very useful, even outside of the problem of plastic deformations due to concentrated forces. As an example, I should like to mention the case of a close fitted pin in a plate (see illustration below). Suppose that it is desired to determine the displacement of the pin with respect to a fixed



boundary circle with radius r_2 under a load on the pin acting in the plane of the plate. If P is the load per inch thickness of the plate E the modulus of elasticity of the material, and r_1 the radius of the pin, the Michell solution gives directly for the displacement the following equation.

$$y = \int_{r_1}^{r_2} \frac{P}{\pi E} \frac{dr}{r} = \frac{P}{\pi E} \log \frac{r_2}{r_1}$$

This is only one of the many examples that may be cited to show the usefulness of this and the other two-dimensional solutions given by the author.

It is hoped that the present paper will point out to engineers some of the many possibilities of these solutions.

JOHN H. HITCHCOCK:[1] Dr. Nadai's excellent paper portrays well the effect of concentrated pressure upon the distribution of stress. In addition to that, the facts brought out in the paper are of special interest to rolling mill engineers in the respect that they offer a new light to the study of the effects of rolling pressure. It has been suspected that the pressure developed in rolling was affected by the ratio of the length of contact between the rolls and the material to the thickness of the material being rolled. Efforts have been made to express this effect quantitatively, but these efforts have been of little value because of the lack of experimental data. Dr. Nadai's paper indicates a method which may be found valuable in further study of this effect, and his results constitute a valuable indication of the nature of its relation. The ratio of the lengths of contact to the thickness of the material rolled in which rolling is commonly done varies from one-fifth to one hundred or more. The observations which Dr. Nadai has made lie in the middle of this range, and it will doubtless be of great value if other observations can be made extending over the entire range of these quantities in which rolling is commonly done.

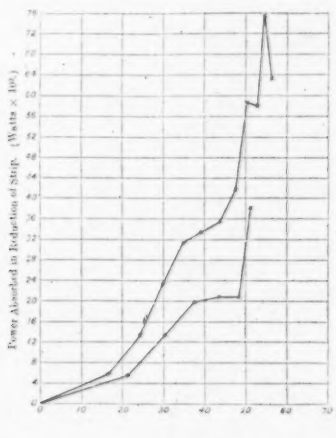
O. W. ELLIS:[2] To anyone who has attempted to take photographs of the type that Dr. Nadai has, it will be of considerable interest to hear more from him regarding the method described on the sixth page of his paper rather briefly. Dr. Nadai submitted to me a number of specimens similar to those which he has described in the paper and asked me if I would produce photographs of the Luders lines which were on the samples. I do not know how many days I spent in trying to get reasonable photographs. I have reason to believe that since that time this new method of illuminating the specimens has produced the very interesting results that are shown in the paper. If Dr. Nadai could give full particulars regarding the method of illumination that he employed, I, for one, should be very grateful to him. On page 1058 there is a remark made which he asked me to raise again because it might be of interest to others here. He says, "It is known that certain ductile metals have the remarkable property of exhibiting a well defined yield point. This means that if the stresses do not reach certain values, the strains remain perfectly elastic and if the yield point is reached, plastic or permanent deformations start to develop." The question raised with Dr. Nadai was that if we accept the drop of the beam as indicating yield point, and I presume that that is what he is referring to, are we to assume from his remarks that he believes that at stresses below that at which the yield takes place no plastic deformation occurs at all. I think that if Dr. Nadai will answer that question in the way in which he answered me it will make that point clear to everyone here.

There is another point about which I asked Dr. Nadai which I think might reasonably be dealt with here. At the bottom of page 1058 Dr. Nadai says, "In the case of a slow stationary plastic flow there exists a relation containing only the components of stress, which is satisfied." The question

[1]Associated with the American Society of Mechanical Engineers, Pittsburgh.

[2]Director of metallurgical research, Ontario Research Foundation, Toronto, Ontario, Canada.

I put to Dr. Nadai was "What is a slow *stationary* plastic flow?" How could a flow be stationary? There is a very definite explanation for that which I am sure Dr. Nadai will be glad to give us. Turning to the points dealt with in the latter part of the paper, the rolling of metals and the effects of cold working on metals have been subjects in which I have always been particularly interested. I think possibly the first experimental results on the amount of energy absorbed in the rolling of brass and copper-nickel were presented in a paper that I gave before the British Institute of Metals



Percentage Reduction in Thickness of Strip, Cast 70:30 Brass.

about 15 years ago. There was, in connection with those tests, a phenomenon first noted by Howe and since noted by many other observers; i. e., the phenomenon of the critical range of plastic deformation. I am wondering whether Dr. Nadai can extend the ideas which he has put forward in this paper and which really, after all, only deal with very small deformations, to cover the large deformations one gets in rolling mills. Take, for example, the rolling of brass. My experience has been that the first reduction is anywhere from 15 to 25 per cent. That is considerably more than anything that is represented in this paper. Even in the example shown in Fig. 52, the reduction is extremely small. Can we assume that theories based on small reductions of that type can be pushed forward to cover a 25 per cent reduction? If I may I will draw upon the board a rough curve showing the relationship between the energy required to deform brass and the percentage deformation such as we found in the experiments referred to above. It will be seen that there is a range—between 35 and 50 per cent reduction—during which the energy required to further deform a piece of brass in the rolling mill—the same is true of copper in the wire mill—*tends* to diminish. Actually, this is associated in some way with the mechanical properties of the material being deformed. In trying to explain this sudden diminution in the amount of energy required to further deform a piece of metal, a number of theories have been suggested. One of them, brought forward by Dr.

F. Johnson of Birmingham, England, is based upon the theory that marked changes in the direction of slip occur during the deformation of such material in the vicinity of the critical range of deformation. I should like to ask Dr. Nadai whether there is any basis upon which we could form such a theory in line with the ideas that he has put forward in this paper. I know that it is a great deal to ask him to reply to at the moment, but it is something which I feel he might, in the future, consider in connection with his experiments along this line.

I feel we are to be congratulated upon having this very interesting paper before us because it brings before this Society a field of investigation to which very little attention has been paid heretofore.

It was because I thought comparatively few people would have access to that journal that I thought Dr. Nadai might be able to draw on the board very rapidly an outline of the optical system that will produce the effects he has shown.

Author's Closure

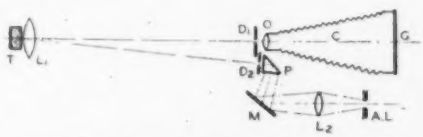
Referring first to the interesting and valuable remarks made by Dr. Ellis, I would like to repeat here some points which my former colleague of the Research Laboratories in Pittsburgh and I have discussed shortly before this meeting. I must agree with Dr. Ellis that in the paper presented there remained several points which, mainly on account of the limitations of space, were perhaps not brought out as clearly as this would have been perhaps desirable. I am, therefore, taking this occasion to add some few remarks which will, perhaps, help to explain further some points treated in the paper.

In one of his questions Dr. Ellis asked the writer regarding a method which was used to observe the slightly deformed surfaces of steel specimens and the thin layers of slip on well polished surfaces of steel pieces just after the yield point has been reached. The method referred to by Dr. Ellis was described in *Schweizerische Bauzeitung*, Zürich, 1924, Volume 83, No. 14 and 15, and may be illustrated by the following sketch (Fig. A) this discussion. The main subject of the illumination method used in the sketch is to photograph the faint phenomena of slip which can be observed on slightly deformed polished metal pieces. The difficulty encountered is to reproduce by a photograph minute details visible on a mirror-like reflecting plane surface. Essential parts in the illuminating arrangement are the large lens L_2 and certain diaphragms located at definite points in the way of the light beam. The test piece T with its reflecting surface turned against the camera C stands behind the lens L_2. It is illuminated by an arc lamp A.L standing beside the optical axis of the photographic camera. By this arrangement one can observe on a screen or on the ground glass G of a photographic camera C the slightest disturbances on a polished surface of a metal piece T by means of reflected light. The method is somewhat similar to that invented by the physical Toepler and called in optics the "Schlierenmethod." For transparent objects it has been used for example to determine the slightest differences in the refracting power of a glass lens, also to photograph, for example, moving air around a flame or to make visible the sound vibrations

in air. (Mach's experiments). In the combination shown in the Fig. A it has been suggested by L. Prandtl and the writer for non-transparent objects.

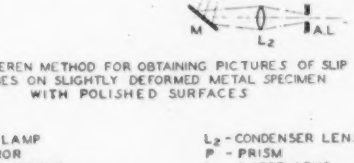
Dr. Ellis asked a question which in a short way might be expressed perhaps as follows: "Are there no plastic deformations under the yield point?" To explain a little better what was assumed with respect to the stress-strain diagram for steel and certain assumptions used Fig. B can be con-

FIG. A



SCHLIEREN METHOD FOR OBTAINING PICTURES OF SLIP LINES ON SLIGHTLY DEFORMED METAL SPECIMEN WITH POLISHED SURFACES

A.L - ARC LAMP
M - MIRROR
D_1 & D_2 - DIAPHRAGMS
T - TEST SPECIMEN WITH POLISHED SURFACE
C - PHOTOGRAPHIC CAMERA
L_2 - CONDENSER LENS
P - PRISM
L_1 - LARGE LENS
O - OBJECTIVE OF CAMERA
G - GROUND GLASS



ACTUAL & IDEALIZED STRESS-STRAIN DIAGRAM FOR MILD STEEL.

FIG. B

sidered. Fig. B may indicate the shape of a tensile stress-strain diagram as obtained in an actual test with a mild steel. It will be observed that this diagram shows only the first portion of this stress-strain curve perhaps to some few per cent of unit elongation and that the stress first increases proportionally with the elongation. At a certain point called the proportional limit the straight line bends into a curve and reaches later a horizontal line; finally the stress stays nearly constant. All this refers only to the stress-strain diagram of a comparatively soft or mild steel. Now this actual diagram can for many purposes be replaced with advantage by an *idealized* diagram as shown in the same figure by the two straight lines. By introducing the broken line ABC as stress-strain diagram, many considerations about the plastic state can be simplified considerably. In this latter and also in the former diagram there is a definite elastic range and a plastic range. In the latter diagram the yield stress stays exactly constant. It is obvious that this is an idealization of the conditions, for example, for the yielding of mild steel and the assumption is true, provided that the deformations do

not exceed certain amounts, (some few per cent). For other materials such as for example, an annealed copper or aluminum, it is well known that the first part of the stress-strain diagram for low values of stress does not show a well defined yield point. In such materials it is first not possible—at least under small stresses—to distinguish between a definite elastic and a definite plastic range. The plastic deformations develop quite gradually with the elastic ones. For such materials as the latter, therefore, the definition of a yield point loses its importance if not much larger elongations are to be considered, under which gradually again an elastic range can be distinguished.

Dr. Ellis asked the writer what was meant under a slow stationary plastic flow. This expression refers to terms used in hydro-dynamics or also in other physical phenomena containing the time as one of the independent variables. In many cases certain quantities, for example, the temperature within a body or the velocity of a moving medium depends upon the variable time. In other cases the velocity at a given point in space may only depend on the coordinates but not on the time as an independent variable. Take, for example, the stretching of a cylindrical bar under pure tension. If the ends (heads of the bar) are moved with a constant speed quite slowly the plastic flow in the bar can be called a stationary flow. The rate of flow would in this case not depend on time. On the contrary, in an impact test, for example, the rate of flow is greatly changing with time. The latter case would be a case of non-stationary plastic flow.

Referring to the observations of Dr. Ellis about a critical range in the power absorbed in the reduction of a rolled strip with the percentage of reduction in thickness, the writer seems in this moment not competent to explain these interesting observations. In several recently published papers, including the one the writer has presented, the complexity of the phenomenon of the rolling of a metal sheet has been demonstrated. Under complexity is meant here that from the standpoint of an engineer or of mechanics it was not yet possible to analyze with sufficient accuracy, for example, the distribution of stresses in the severely stressed parts of the rolled sheet; or for example, to predict how these stresses would change with the width of the area of contact of the rolls and the sheet or how the stresses will depend on the velocity of rolling. A further difficulty arises if large reductions in thickness are considered. A more exact analysis of the deformations is in this latter case considerably more difficult as in the case of quite small deformations or reductions in thickness as considered by the writer. On account of this and other factors not mentioned here, which might have an influence on the critical power range of rolling a metal sheet after large reductions which was demonstrated by Dr. Ellis, it can be hoped that such valuable observations may stimulate the need of further research work on this question until the factors which affect the force required to roll sheets under given conditions will be better known.

Referring to the remarks of Mr. John H. Hitchcock, the writer is also of the opinion that an important factor on which seems to depend the force required to roll a metal sheet is the ratio of the width of the area of con-

tact between rolls and sheet to the thickness of the sheet. The longer the area of contact, that is, with other words expressed, the greater the diameter of the rolls is compared to the thickness of the sheet, the more the friction between the rolls and sheet will come in action and will tend to prevent the lateral plastic flow within the sheet. The smaller the area of contact is or the smaller the diameter of the rolls, the less secondary friction will be produced in the surface of contact.

Referring finally to Mr. Soderberg's valuable remarks, the writer has endeavored to show that certain investigations which were made under the assumption of purely elastic deformations, might prove to yield also perhaps useful information, if the limit of plasticity has been reached. It is true that it will need greater precautions if such informations will be generalized to cases, for which they were originally not intended to be applied and further investigations will be necessary where the elastic and the plastic parts of the deformations in mechanics become of the same order of magnitude. Mr. Soderberg's short remarks should be appreciated on account of pointing to other valuable applications of similar stress distributions to engineering problems.

RESISTANCE OF STEELS TO ABRASION BY SAND

By Samuel J. Rosenberg

Abstract

A study has been made of the adaptability of three types of apparatus, namely, the ball mill, the sand blast, and the Brinell machine, to the testing of the resistance of metals to abrasion. In all of these tests wear was caused by the abrasion of sand on metal and not by metal on metal.

In the ball-mill test 1-inch spheres of the material tested were tumbled in sand for definite periods. This test proved to be generally unsatisfactory as a laboratory test.

In the sand-blast test a specially designed nozzle directed a stream of air and sand at definite pressures against a specimen of the material, 3 inches square by ¼-inch thick, held at an angle of 45 degrees to the axis of the nozzle. This test gave excellent check results but did not differentiate between many of the materials tested which were known to have widely different wear-resisting characteristics. This failure to differentiate was apparently due to the fact that the abrading conditions were not sufficiently severe, as was evidenced by the fact that some differences noticed with an air pressure of 60 pounds per square inch were not noticed at a pressure of 30 pounds per square inch.

In the Brinell test the plane surface of a specimen was pressed under a definite load against the edge of a slowly rotating open-hearth iron disk while a constant stream of standardized, fine-grained sand was fed between the surfaces of the disk and the specimen. This test was found to be generally satisfactory.

A series of carbon steels, varying from 0.08 to 1.29 per cent in carbon content, was tested in the normalized, annealed, and hardened conditions. The results of these tests showed that the resistance to wear of these steels in this special type of test was dependent upon the hardness. A study of the effect of tempering temperature

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upon the wear showed the same trend, i. e., the resistance to wear increased with the hardness.

The effect of grain size upon the resistance to wear was studied by testing a 0.20 per cent and a 0.76 per cent carbon steel annealed at various temperatures. An increased temperature of annealing resulted in a general increase in resistance to wear as evidenced by a decreased loss of weight, this effect being more marked in the low-carbon steel than in the high-carbon steel.

A study of the mechanism of wear or abrasion showed that grains of the steel were not torn off in their entirety but were broken into small particles, this action being accompanied by local strain hardening.

I. Introduction

THE problem of testing the resistance of metals to wear is one of the most perplexing problems confronting the testing engineer today. So many different variables enter into the resistance of metals to wear that it is generally agreed that there is no universal test for determining this resistance. Attempts have been made, therefore, by various investigators, to study certain types of wear with machines especially designed to simulate certain conditions of service, and this method seems to be the most promising mode of attack.

The object of this investigation was not to find a universal testing machine for all conditions of service, because the development of such a machine does not appear to be probable, but it was believed that closely controlled laboratory tests with a series of ferrous metals would be of value as a contribution to the general problem of the testing of the resistance of metals to abrasion.

Wear may be divided into two major types—wear caused by abrasion of metal on metal, and wear caused by abrasives on metal. The latter type only will be considered in this paper.

II. Previous Investigations

Probably the earliest work on abrasive wear was carried out by Robin.[1] His tests consisted in determining the wear of steels

[1]Felix Robin, "The Wear of Steels with Abrasives," Carnegie Scholarship Memoirs, Iron and Steel Institute, II, p. 6; 1910. Also "Abrasion Tests of Steel," International Association for Testing Materials, Sixth Congress, IIIa; 1912.

when rubbed under a known pressure on papers which were covered with an abrasive powder. A cylindrical steel specimen (50 mm. in diam.) described a circular path having a diameter of 150 mm.; the pressure used was 1 kg. per sq. cm., and the speed was 70 revolutions per minute. The abrasion number was the sum of the weights of metal lost by abrasion in three consecutive tests, each lasting a minute.

For the same type of steel with the same abrasive, two sheets of different papers gave comparable results. Precision was from 2 to 10 per cent on ordinary steels and from 15 to 20 per cent for very hard steels. The effect of variables was stated to be as follows:

"Pressure—Augments the wear approximately proportionately if within 0.5 to 2 kg. per sq. cm. In hard steels the wear increases at a more rapid rate and the curve is parabolic. The wear is proportional to the surface area of the specimen.

"Speed—Increases the abrasive power of the paper. Some steels are more sensitive than others, especially those which were also sensitive to variations in pressure.

"Nature of abrasive—Different abrasive materials and papers of different sizes of grain class the metals in approximately the same order while they give different figures as functions of the abrasive power.

"Wear of steels—In the case of annealed carbon steels, the wear is not proportional to the pearlite. A maximum of abrasion was found with 0.4 per cent carbon; in hypereutectoid steels the wear was approximately proportional to the carbon percentage. Increased fineness of the particles, cold work, and the presence of phosphorus increase the resistance to abrasion; the presence of silicon and manganese diminish it frequently. In cast metals the resistance grows with the phosphorus contents and with the percentage of iron carbide. The hardened steels can hardly be distinguished by this process; on the contrary, tempered steels lend themselves to this examination without any difficulty. The best resistance in hardened steels seems to be characteristic of the finest martensites.

"The practical conclusions to be drawn for the domain of the science of testing are the following:—each mode of measurement and each kind of test leads to a special classification of the steels. The diverse modes of determining the wear cannot be

estimated by one and the same test. The test which has been explained so far appears only to lend itself for the examination of rails under certain special service conditions, to files, and to apparatus for disintegration, etc."

Brinell[2] tested a variety of materials for resistance to wear using a machine designed by him and described elsewhere in this report. The effect of variables was stated to be as follows:

"1. Coarse-grained sand produces greater wear than finer sand under equal conditions.

"2. In using pure quartz sand, wear is greater than that produced by ordinary sand.

"3. Majority grain size of sand is of decisive influence as to abrasive action, therefore, two sorts of sand with equal size of smallest and largest grain may possess a widely different abrasive effect.

"4. Grain form is not of influence, at least at pressures up to 10 kg., but in the investigation of softer materials at lower pressures the sand with sharp corners produces a greater wear than sand of round grain."

In the carbon steels he found that resistance to wear increased with hardness.

A machine for the determination of abrasive wear was described by Parker.[3] In making a test the specimen was clamped with a definite force for a definite length of time against the face of an alundum grinding wheel, which was continuously dressed. The number of revolutions required to produce a given depth of wear was recorded automatically. The only results reported showed that of three materials, one untreated, one with a thin carburized case, and one with a thick carburized case, the last mentioned had the greatest resistance to wear, with the thin carburized case running a close second. The untreated material had a relatively low resistance to wear.

A machine for determination of wear under wet grinding con-

[2]J. A. Brinell, "An Investigation of the Resistance of Iron, Steel and of Some Other Materials to Wear," *Jernkontorets Annaler*, 1921. A more convenient reference is the transcript by H. A. Holz, "Brinell's Researches on the Resistance of Iron, Steel and Some Other Materials to Wear," *Testing*, I, p. 104; 1924.

[3]W. H. Parker, "The Wearing Qualities of Tire Chains," *Proceedings*, American Society for Testing Materials, Vol. 28-2, 1928, p. 332.

ditions was described by Blake.[4] In principle, the machine was an accurately controlled grinding machine in which all conditions as to abrasive, pressure, speed, time, and moisture were under control. The wear was measured by the amount of metal worn off in approximately 15,000 feet of travel at a speed of 125 feet per minute, with a pressure of 50 pounds per square inch on a testing face of approximately 3 square inches, and was expressed as the ratio of loss of weight of the metal tested to loss of weight of an adopted standard (commercially pure iron). His conclusions are:

"This abrasion tester has shown that each abrasive or mineral substance develops its own order of relative abrasion resistance for metals and that the position of any steel or alloy on a listing of abrasion factors will depend upon the nature of abrasive employed. In extreme cases it has been found that steels at the top of the list with one abrasive are at the bottom of the list with another. This discovery has eliminated the idea of securing general all-around high abrasion resistance in any one steel."

An actual service test was made by Clapp and Deveraux[5] in which six 5-inch diameter chromium steel balls and six 5-inch diameter chromium-molybdenum steel balls were placed in a ball mill. The material ground was 3-inch maximum size chalcopyritic ore in which the gangue was a hard shale and quartz. After six weeks the chromium steel balls were 2 to 2½ inches in diameter while the others were 4 to 4½ inches in diameter.

III. Equipment and Test Methods

1. Ball-mill Test

The mill used for this test consisted of two cylindrical containers which revolved about their longitudinal axes during test at a speed of 55 revolutions per minute. The interior of the container used in the tests (only one mill was used) was lined with rubber to prevent the impinging of the steel specimens upon the porcelain walls. After being so lined the size of the container was 9 inches

[4] J. M. Blake, "Wear Testing of Various Types of Steels," *Proceedings*, American Society for Testing Materials, Vol. 28-2, 1928, p. 341.

[5] E. C. Clapp and F. C. Deveraux, "Tests with Molybdenum Steel Balls at Matahambre, Cuba," *Engineering & Mining Journal Press*, Vol. 120, 1925, p. 891.

in diameter by 12 inches long. Fig. 1 shows a photograph of the apparatus.

The specimens used for this test were ground and lapped into 1-inch spheres. Ten kilograms of a certain sand was placed in the mill at the beginning of each test run, the samples were then laid in the sand, the cover tightened, and the mill started. Entirely new sand was used for each test. The diameters of the specimens were carefully measured and the losses of weight determined after each run. Wear was expressed as loss of weight (milligrams) per square centimeter of surface per hour of test.

2. *Sand-blast Test*

Since the sand blast is such a rapidly abrading process it seems logical to attempt to adapt it to abrasion testing. Fig. 2 is a diagrammatic sketch of the apparatus designed and used for this test. In this test a special nozzle, 5/16 inch in diameter, was placed in the top of a rubber-lined wooden box. The nozzle was connected to the bottom of a Y pipe fitting. Sand and air were introduced through the two upper branches of the Y, the air passing through an inner tube into the Y. The lower end of this tube, which carried the air blast into the nozzle, was below the point at which the sand was introduced so as to avoid "backing up" of the sand. The introduction of the sand was controlled by a slide valve.

The specimens used were flat, 3-inch squares, about ¼ inch thick, held in place at an angle of 45 degrees to the axis of the nozzle and about 3½ inches below it. An opening in the bottom of the box provided an outlet for the sand and air.

In all tests a weighed amount of sand was placed in the sand container. A rubber hose connected the bottom of this container with the sand inlet. The air blast was adjusted to the proper pressure, and a slide valve in the sand inlet was opened, thus giving an immediate and full flow of sand. The air blast, emerging from the tube, helped suck the sand downward and automatically caused mixture in the nozzle and abrasion of the specimen.

The specimens were cleaned and weighed before and after test and the loss of weight taken as a measure of the resistance to wear. Several consecutive tests were made on each specimen until a definite rate of wear was observed.

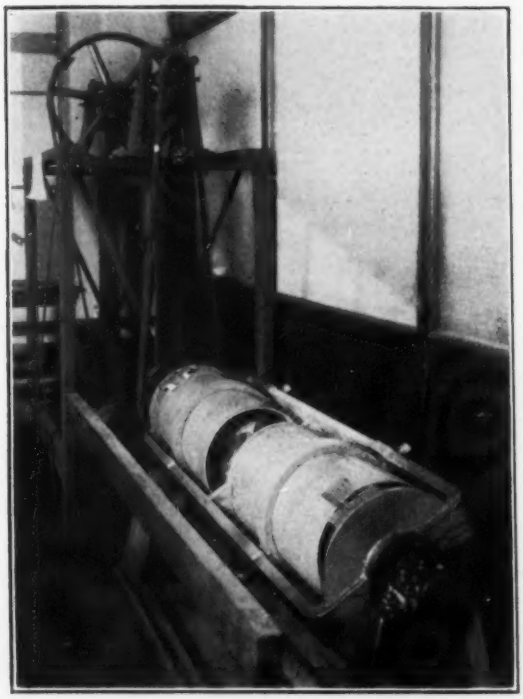


Fig. 1—Photograph of Ball Mill.

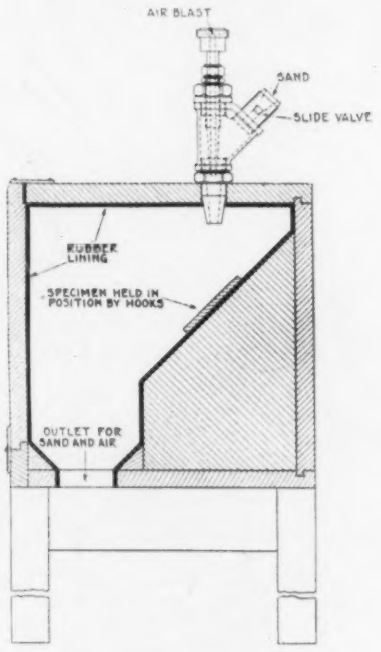


Fig. 2—Diagrammatic Sketch of Sand-blast Apparatus.

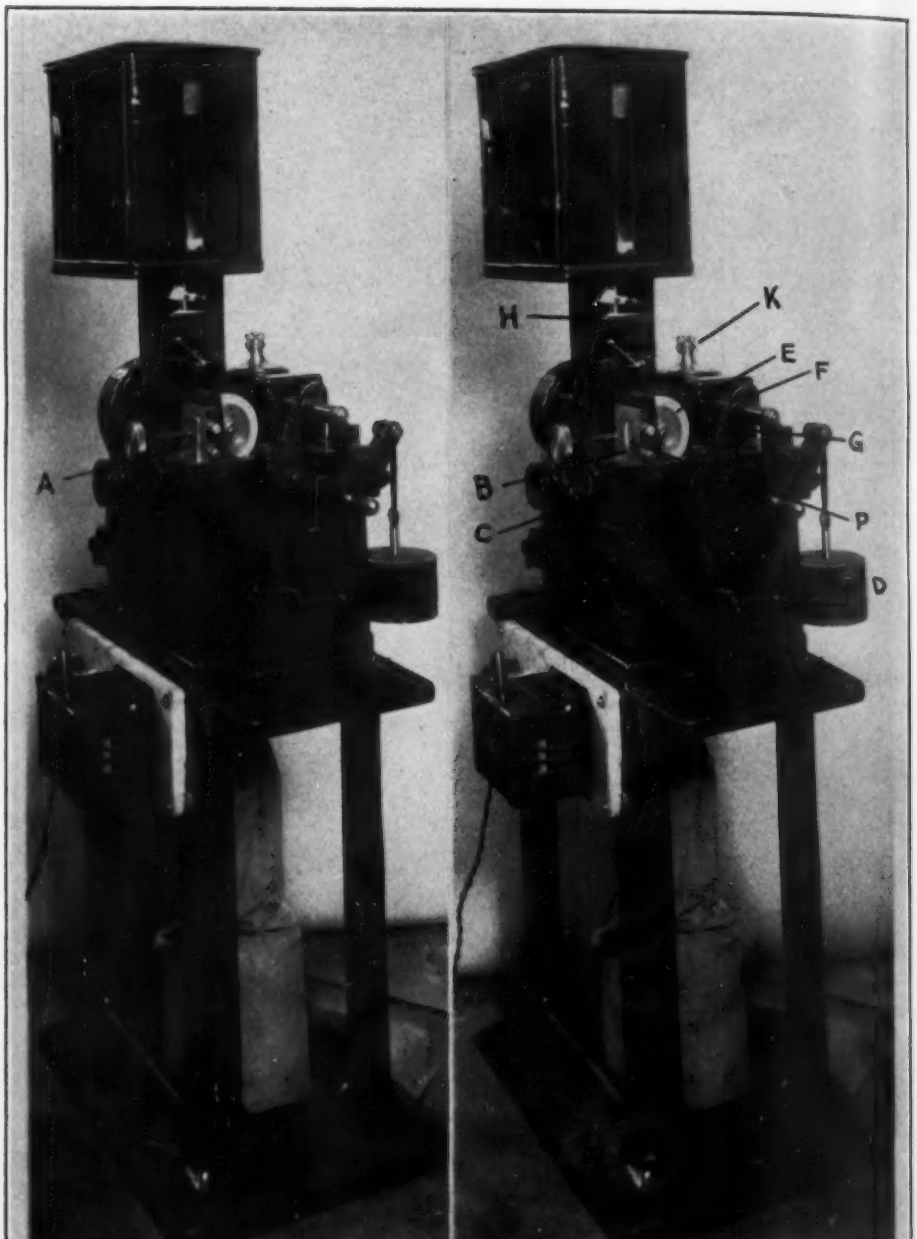


Fig. 3a—Photograph of Brinell Machine with Specimen in Position for Test. Fig. 3b—Photograph of Brinell Machine with Specimen Removed.

3. *Brinell Test*

A photograph of the Brinell machine for testing the resistance

of materials to wear, with and without a specimen in place, is shown in Figs. 3a and 3b. In operating the machine, the specimen, A, was clamped to a slotted plate, B, mounted on a carriage, C, to which was attached a cord passing over a pulley and carrying the weights, D. A disk, E, of open-hearth iron, 100 mm. in diameter and 4 mm. thick, was so mounted on a shaft that the center line of its face

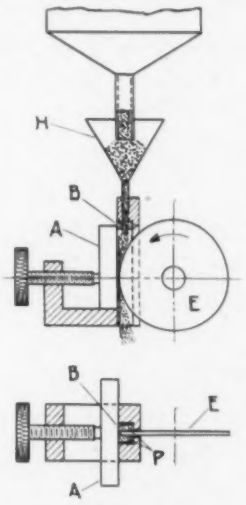


Fig. 4 — Diagrammatic Sketch of Essential Parts of Brinell Machine.

coincided with the center line of the slot, B, against which the specimen was clamped. When the cam, F, working against a stop, G, on the slide was turned to the proper position, the carriage, C, mounted on ball bearings, moved to the right until the specimen rested against the abrasion disk, the pressure between the two being determined by the weights attached to the end of the slide.

A hopper, H, mounted above the slotted plate was filled with sand fed into it from the receptacle placed above. During the test a continuous stream of sand was passed between the specimen and the disk. The disk had a speed of 45 revolutions per minute, and the linear travel of a point on its circumference could be adjusted by a graduated wheel and locknut, K, on top of the machine. After the disk had run the pre-determined distance, the motor stopped automatically. During the test the motor also actuated a small fan which sucked the used sand down into a bag. Fig. 4 is a diagrammatic sketch illustrating the essential part of the machine.

The principle of the test, then, is as follows:—The plane surface of the test specimen is pressed under a definite load against the edge of a slowly rotating disk while a constant stream of standardized, fine-grained sand is fed between the surface of the disk and the specimen. After the circumference of the disk has run through the distance required for a test, the specimen is removed, washed, dried, and weighed, and the maximum depth of the segmentary groove produced is measured by means of a depth indicator reading directly to .01 mm. The wear-resistance number "Nm" is calculated from the formula $Nm = \frac{1000}{A}$, where A is the volume in cubic millimeters of the worn-out segment for 1-mm. thickness of the disk. In tables furnished with the machine values of Nm are given for different values of diameter of disk and maximum depth of groove.

Two methods of expressing the resistance to wear as determined by the Brinell machine are (1) by means of the "Nm" numbers and (2) by means of the losses of weight. The former is rather unreliable and frequently gives erratic results, due to the difficulty of securing an accurate reading of the depth of the groove because of the relatively rough bottom. This error is aggravated by the human equation in reading the dial, the pointer of which wavers over an appreciable distance as the point of the gage is moved but slightly over the rough bottom. For these reasons, although the "Nm" numbers are given on the curves, (Figs. 9 and 13) the losses of weight have been used as the criterion of the rate of wear (or resistance to wear).

In the tests made, none of the abrading disks was used when its diameter wore below 97.5 mm. When a new wheel was used for the first time, its surface was roughened by first running it against a dummy specimen in the machine. During a test the edge of the disk often burred over, especially when the test specimen was of soft steel. In these cases the faces of the disk were dressed by a file after each test.

Inconsistent results were at first obtained by this machine. By sieving the sand used and by removing the two guide plates, P (Fig. 4), thus allowing a free flow of sand, results were secured which gave good checks.

IV. Results

1. Ball-mill Test

Two series of tests were made. In one, standard Ottawa silica sand, 20-30 mesh, was used, while in the other, Illinois glass sand, a much finer and sharper sand, was used. The results of these tests, together with the compositions and heat treatments of the steels tested, are summarized in Tables I and II. All specimens marked "C" were cast, while specimens marked "F" were forged. Widely different test results were usually obtained on the same material during different tests. The reason for this is not at all clear.

In order to obtain some idea of the trends shown by the tests, the last ten 24-hour runs with each abrasive were averaged. The differences between the various steels are not very marked. It is of interest to note that the forged and hardened specimens in each group lost the least amount of weight. Generally speaking, the .97 per cent chromium steel was found to be the most wear-resistant steel in the series. Contrary to what might have been expected, in this type of test the finer, sharper sand (Illinois glass sand) caused less wear than the rounded Ottawa sand.

2. Sand-blast Test

Fig. 5 shows the results of tests on a series of steels together with the compositions and heat treatments. Plotting the losses of weight against the amount of sand blasted on the specimens gave, after a wearing-in period, a straight-line relationship. The loss of weight of the specimen per unit amount of sand blasted was determined from the slope of the line. It was surprising to note that all the different steels, even when subjected to different heat treatments, showed about the same rate of wear. The very slight increase in rate of wear shown by Heats No. 85046HM and 68290HM was not due to the steel but to the use of a new sand-blast nozzle as will be explained later.

The results of tests on a series of 0.45 per cent carbon steels, to show the effects of additions of Ni, Cr, and V as alloying elements, are shown in Fig. 6. The sand used in these tests was different from the sand used for the tests summarized in Fig. 5, therefore no obvious correlation between the two sets of curves is to be

Table I
Results of the Ball Mill Tests

(Standard Ottawa Silica Sand, 20-30 mesh).

Specimen	Composition	Condition	Unit Loss—Mg. per sq. cm. per hr. x 10^3 15 hrs.	15 hrs.	15 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	Av. last 10 24 hr. runs
0C1	.41 C, 3.04 Ni,	Normalized at 815°C.	11.3	11.0	11.6	12.9	10.8	9.6	8.1	7.5	13.9	10.2	12.1	12.9	11.0	11.4	10.7
0C3	.72 Mn, .13 Si	Normalized at 815°C.	13.3	8.8	12.0	13.9	12.1	9.8	8.7	6.9	12.5	10.0	11.4	13.7	10.6	11.4	
0F1		Normalized at 815°C.	2.0	8.9	10.9	15.2	12.4	10.1	7.4	8.0	14.8	10.7	10.7	12.4	12.1	11.8	10.5
0F2		Normalized at 815°C.	9.6	11.2	15.2	13.8	13.2	9.5	8.4	6.2	15.6	10.7	9.9	13.0	10.3	11.9	
0F3		Normalized at 815°C.—	13.2	11.8	15.5	14.8	9.8	10.1	5.8	6.8	10.5	9.9	10.9	12.5	10.3	10.7	9.6
0F4		oil-quenched from 815°C. and drawn at 100°C.	12.8	11.5	10.5	16.2	9.8	9.7	6.0	6.8	11.1	9.7	10.7	11.1	9.7	9.7	
1C1	.46 C, .97 Cr,	Normalized at 815°C.	9.3	9.3	11.6	13.1	10.2	8.5	6.9	6.7	12.5	8.5	9.8	12.7	9.8	10.6	9.6
1C3	.75 Mn, .14 Si	Normalized at 815°C.	18.9	10.6	12.3	13.1	11.2	8.1	8.3	5.4	11.5	8.3	11.8	11.2	9.1	10.4	
1F1		Normalized at 815°C.	15.8	10.2	10.9	14.2	10.3	8.2	6.8	5.8	13.0	9.3	10.3	11.8	9.9	10.7	9.5
1F2		Normalized at 815°C.	19.9	10.3	11.0	14.7	9.8	9.4	7.5	6.2	11.1	8.9	10.0	12.1	9.4	9.3	
1F3		Normalized at 815°C.—	5.9	9.5	10.9	16.0	9.5	8.8	5.4	6.6	10.1	9.5	9.5	11.5	8.9	10.3	8.8
1F4		oil-quenched from 815°C. and drawn at 200°C.	9.3	10.3	10.6	14.9	8.7	8.5	4.8	6.4	8.9	9.5	10.0	10.6	9.8	9.3	
3C1	.43 C, .45 Mn,	Normalized at 815°C.	22.0	12.3	24.7	15.4	14.9	11.0	10.6	9.6	15.2	14.6	12.7	16.0	12.5	12.1	12.6
3C3	.09 Si	Normalized at 815°C.	18.9	12.9	14.6	16.2	15.2	10.6	7.9	9.5	14.9	11.4	11.2	16.0	11.6	13.1	
3F1		Normalized at 815°C.	17.0	11.0	20.3	15.2	13.3	10.4	10.2	8.8	15.2	11.0	13.3	12.1	11.9	13.1	11.9
3F3		Normalized at 815°C.— brine-quenched from 815° C. and drawn at 200°C.	12.6	10.6	12.6	14.5	9.3	8.9	5.0	6.8	10.0	9.3	10.2	11.0	9.4	10.0	9.0
4C1	.46 C, .26 V,	Normalized at 815°C.	24.8	11.7	14.7	14.6	13.0	10.5	7.3	7.3	15.7	11.5	14.4	13.1	13.4	13.4	11.8
4C3	.71 Mn, .12 Si	Normalized at 815°C.	16.0	12.3	17.6	12.5	13.3	10.2	8.1	8.7	16.0	10.8	10.4	13.7	12.3	12.5	
4F1		Normalized at 815°C.	+	8.6	12.6	13.1	11.2	8.9	7.5	7.1	12.7	9.8	10.4	14.5	11.0	10.4	10.6
4F2		Normalized at 815°C.	13.9	9.6	13.3	12.2	11.0	9.5	7.9	7.7	13.1	11.4	10.4	13.1	12.9	10.8	
4F3		Normalized at 815°C.—	9.3	11.6	8.6	14.7	10.4	8.3	5.8	6.4	9.8	8.9	10.4	10.6	9.6	10.2	8.8
4F4		brine-quenched from 815° C. and drawn at 200°C.	11.3	11.0	10.3	14.3	8.7	8.3	5.8	6.4	9.6	8.5	10.4	8.9	8.9	9.6	
5C1	.43 C, .46 Mo,	Normalized at 815°C.	14.0	11.0	11.0	13.6	10.8	9.6	7.9	6.5	15.0	10.4	11.2	11.9	10.6	12.1	10.8
5C3	.64 Mn, .13 Si	Normalized at 815°C.	14.3	10.0	12.3	14.5	13.1	10.0	7.7	6.6	13.9	10.4	11.0	12.4	10.8	13.1	
5F1		Normalized at 815°C.	28.5	14.6	24.5	15.3	13.0	10.8	8.1	7.7	14.1	11.6	11.4	14.9	12.4	11.4	11.5
5F3		Normalized at 815°C.— oil-quenched from 840°C. and drawn at 300°C.	14.6	11.0	13.3	14.5	9.8	9.6	6.0	7.1	10.6	9.6	10.2	11.4	9.6	9.1	9.3

Rockwell "C" hardness of all heat-treated samples was approximately 50.

Table II
Results of the Ball Mill Test

Table II

Results of the Ball Mill Test

(Illinois Glass Sand).

Specimen	Composition	Condition	Unit Loss—Mg. per sq. cm. per hr. x 10^3												
			24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	24 hrs.	Av. last 10 runs
0C1	.41 C, 3.04 Ni,	Normalized at 815°C.	5.5	7.3	7.1	5.1	6.1	10.0	11.4	11.2	7.9	5.6	4.9	3.7	7.5
0C3	.72 Mn, .13 Si	Normalized at 815°C.	6.9	7.9	4.4	8.0	6.9	11.4	9.4	10.2	10.2	5.0	5.3	5.2	
0F1		Normalized at 815°C.	5.8	7.2	3.5	7.9	6.6	14.0	13.0	12.0	6.0	5.8	4.8	4.9	7.8
0F2		Normalized at 815°C.	5.4	7.2	5.1	8.5	5.0	10.0	13.0	11.1	7.2	6.0	4.8	6.8	
0F3		Normalized at 815°C.—oil-quenched	3.7	6.0	4.7	6.1	3.4	11.8	6.8	7.2	4.5	4.9	4.8	4.9	5.9
0F4		from 815°C. and drawn at 100°C.	2.9	5.2	3.3	5.9	4.2	7.2	13.4	8.2	4.9	4.1	5.2	2.9	
1C1	.46 C, .97 Cr	Normalized at 815°C.	2.5	5.6	5.0	6.1	3.2	10.2	10.6	8.3	7.3	4.0	4.3	7.3	6.4
1C3	.75 Mn, .14 Si	Normalized at 815°C.	3.3	6.9	4.4	6.6	4.6	10.4	6.2	9.1	6.7	5.0	3.9	4.8	
1F1		Normalized at 815°C.	4.9	4.3	3.9	6.3	5.0	8.4	9.7	9.1	6.0	4.9	6.2	4.7	5.8
1F2		Normalized at 815°C.	2.5	6.2	2.9	6.4	4.4	8.7	7.5	8.7	2.7	4.2	4.3	2.5	
1F3		Normalized at 815°C.—oil-quenched	2.1	4.5	5.8	4.5	4.0	7.4	10.1	6.6	4.9	3.5	4.8	4.9	5.2
1F4		from 815°C. and drawn at 200°C.	1.7	4.8	5.0	4.1	4.0	7.5	7.1	5.4	4.2	3.7	3.7	3.1	
3C1	.43 C, .45 Mn,	Normalized at 815°C.	6.0	8.5	6.7	10.3	7.9	12.3	14.2	10.4	9.6	7.3	8.4	5.0	8.8
3C3	.09 Si	Normalized at 815°C.	6.0	7.7	5.6	10.0	6.7	11.8	10.6	11.6	9.3	7.5	4.9	6.2	
3F1		Normalized at 815°C.	7.1	9.4	5.4	7.8	7.5	11.0	10.8	11.9	7.3	6.0	6.7	7.5	8.2
3F3		Normalized at 815°C.—brine-quenched from 815°C. and drawn at 200°C.	2.3	6.0	3.7	4.9	4.2	8.3	6.6	6.2	6.2	3.3	4.1	3.7	5.1
4C1	.46 C, .26 V	Normalized at 815°C.	6.1	8.6	5.6	7.2	6.7	9.6	11.1	11.3	5.6	5.9	4.9	3.7	7.4
4C3	.71 Mn, .12 Si	Normalized at 815°C.	4.0	6.9	5.2	6.1	5.5	11.6	9.4	10.2	9.1	6.6	6.5	5.4	
4F1		Normalized at 815°C.	4.2	8.1	6.0	7.2	5.1	12.7	11.6	10.6	7.7	6.0	5.1	4.6	7.6
4F2		Normalized at 815°C.	4.8	7.7	7.1	5.9	6.1	14.3	11.8	8.3	7.5	6.0	5.9	3.5	
4F3		Normalized at 815°C.—brine-quenched from 815°C. and drawn at 200°C.	2.3	6.2	4.6	5.3	4.0	6.7	4.4	8.9	5.0	3.5	3.7	4.4	5.1
4F4			1.9	6.0	3.7	4.1	4.4	8.9	6.4	7.5	5.4	4.0	4.5	2.1	
5C1	.43 C, .46 Mo,	Normalized at 815°C.	3.8	8.7	2.7	8.0	6.3	9.6	11.2	10.2	5.6	8.3	6.1	6.4	7.3
5C3	.64 Mn, .13 Si	Normalized at 815°C.	5.0	6.7	5.0	7.2	6.3	9.5	8.1	11.0	8.9	4.2	4.5	6.0	
5F1		Normalized at 815°C.	6.8	8.7	4.8	6.9	5.4	13.9	9.1	9.1	9.1	6.2	3.4	4.8	7.3
5F3		Normalized at 815°C.—oil-quenched from 840°C. and drawn at 300°C.	2.9	5.2	6.7	6.6	4.0	10.8	6.4	8.5	5.6	5.4	5.1	4.2	6.3

Rockwell "C" hardness of all heat-treated samples was approximately 50.

expected. Here, again, it was found that differences in composition and in heat treatment were not reflected by the resistance to wear, all steels showing the same rate of wear well within the limit of experimental error. The sand used in this test was standard Ottawa silica sand, 20-30 mesh in size, at an air pressure of 60 pounds per

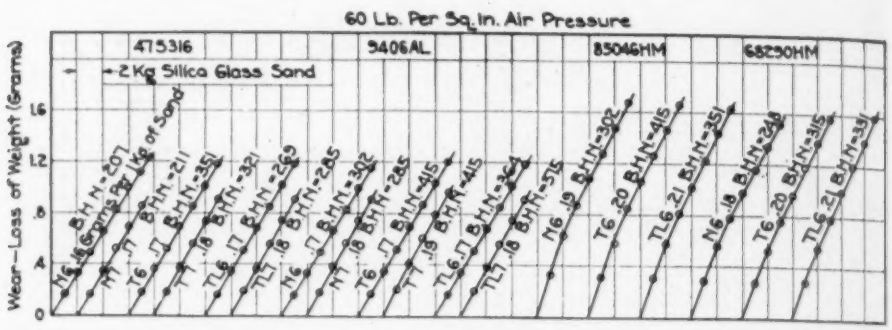


Fig. 5—Resistance to Abrasion of Several Steels as Determined by the Sand Blast.

Heat Number	Per Cent Carbon	Per Cent Manganese
47S316	0.64	0.53
9406AL	0.70	1.13
85046HM	0.63	1.79
68290HM	0.44	1.89

Heat Treatment

N—Normalized at 870 degrees Cent. (1595 degrees Fahr.)

T—Normalized at 870 degrees Cent. (1595 degrees Fahr.) Quenched in Oil from 830 degrees Cent. (1525 degrees Fahr.) and Tempered at 425 degrees Cent. (795 degrees Fahr.)

TL—Normalized at 870 degrees Cent. (1595 degrees Fahr.) and Quenched in Lead at 400 degrees Cent. (750 degrees Fahr.) From 830 degrees Cent. (1525 degrees Fahr.)

square inch, and the loss of weight per 2 kg. of sand blasted was about 0.095 gram.

Sand-blast tests on some widely different materials are summarized in Fig. 7, both at 30 and 60 pounds per square inch pressure. The losses of weight when tested with the 30-pound pressure were considerably less than when tested under the 60-pound pressure, but the order of rates of wear was the same for the specimens.

In the tests under the 30-pound blast, no difference in resistance to wear was apparent between the two chromium-molybdenum steels, the 12 per cent manganese steel, and the white iron. Open-hearth iron showed a slightly higher loss of weight while gray iron lost considerably more.

The tests made under the 60-pound blast showed that there was no difference between the two chromium-molybdenum steels and the 12 per cent manganese steel. The white iron, however,

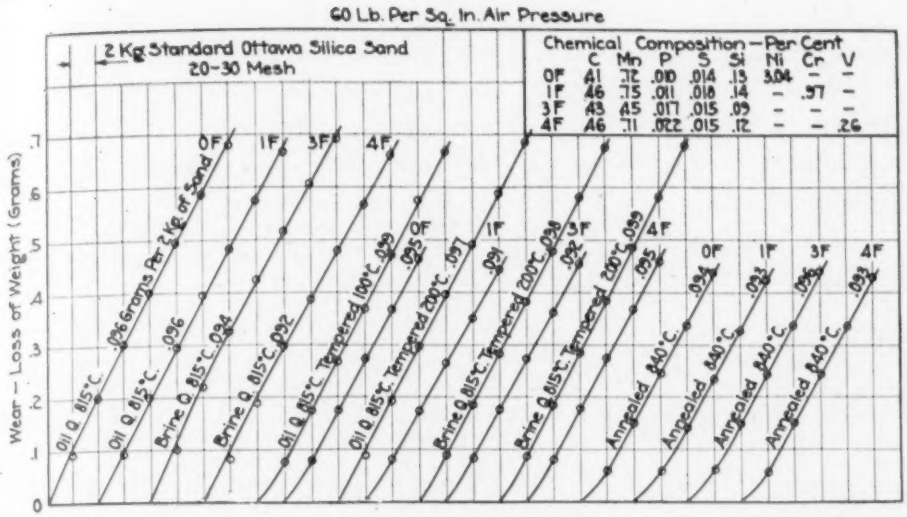


Fig. 6—Resistance to Abrasion of Several Steels as Determined by the Sand Blast.

showed a decidedly higher loss of weight, the open-hearth iron showed still more, and the gray iron again lost considerably more than any of the others.

It is apparent from the curves in the three figures just given that the sand-blast is an extremely accurate test. As long as the same conditions of test existed, duplicate tests gave results which checked better than would be expected. The main variable difficult to control was the diameter of the sand-blast nozzle. This nozzle gradually wore and so enlarged, and as the area of the nozzle increased, the losses of weight of the test specimens decreased. It was, therefore, imperative to replace this nozzle at frequent intervals.

The fact that this test failed to differentiate between various steels indicated that sensitivity was lacking. It is probable that the test conditions used were not proper to bring out large differences in resistance to wear that may be inherent in the materials tested, and this reasoning is supported by a further study of the results.

If two materials such as chilled iron and gray iron be subjected to a light flow of sand under no pressure other than the force of gravity, it would not be surprising to find that one was about as resistant to abrasion as the other. As the velocity of the blast of sand increased, however, the chilled iron would begin to show its inherently higher abrasive-resistant qualities, and as the blast became still more severe, the difference between the two would probably become more and more apparent.

On applying this theory to the results shown in Fig. 7 at least one difference is apparent, namely, the increased relative loss in weight of the white iron to the steels at the higher pressure. It appears logical, therefore, to reason that the severity of the test is still not great enough to bring out the differences in resistance to wear inherent in all the materials tested.

The sharpness of the sand is also of importance in determining the rates of wear of materials. It should be noted from results

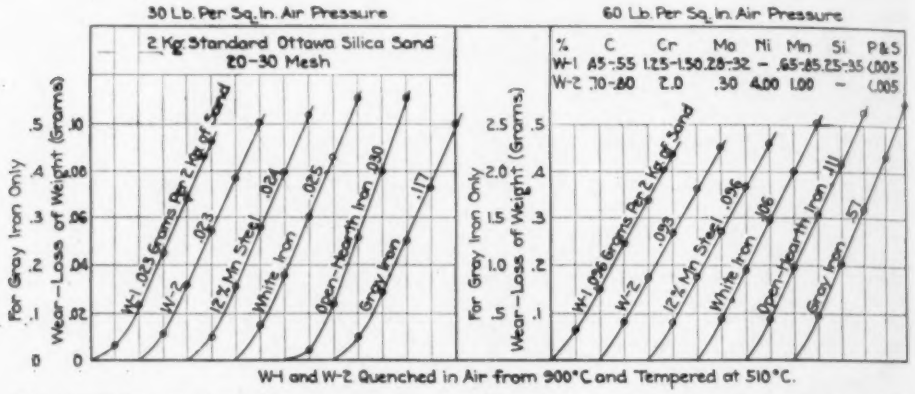


Fig. 7—Effect of Sand-blast Pressure on the Resistance to Abrasion of Some Materials.

shown in Figs. 5 and 7 that when the sand blast was used as the abrading medium, sharp sand (Illinois glass) produced rates of wear which were about four times those obtained on similar specimens tested with larger and more rounded sand (Ottawa) at constant pressure.

These results show that many factors affect the rates of wear with this type of test, so it would be expected that slight changes in actual conditions of service would affect the rates of wear of materials considerably.

3. Brinell Test

a. Selection of Sand

The sand used as the abrading medium in the Brinell test was Illinois glass sand[6] passed through a sieve of .297 mm. opening. It was found necessary to sieve this sand because of its variation in grain size. This size of sand was considerably smaller, incidentally, than the size used by Brinell who recommended a grain size of 0.2 to 0.8 mm. The use of sand of such wide limits in size in-

[6]Furnished through the courtesy of the Tavern Rock Sand Co., Toledo, Ohio.

Table III
Comparison of Dried and Undried Sand as Abrasives in the Brinell Test

Duration of Test	Weight of sand passed through Sand not dried	Weight Loss grams	Nm No.
4 min. 9⅖ sec.	4 lb. 5¾ oz.	.1504	190
4 min. 10 sec.	4 lb. 5¾ oz.	.1443	207
4 min. 11 sec.	4 lb. 5¾ oz.	.1471	201
4 min. 18 sec.	4 lb. 7¾ oz.	.1528	196
Av. 4 min. 12 sec.	4 lb. 6¼ oz.	.1486	198
	Sand as dried		
4 min. 14 sec.	4 lb. 9 oz.	.1380	213
4 min. 11 sec.	4 lb. 7½ oz.	.1362	213
4 min. 11 sec.	4 lb. 7½ oz.	.1332	227
4 min. 10 sec.	4 lb. 6¾ oz.	.1349	220
Av. 4 min. 11 sec.	4 lb. 7¾ oz.	.1356	218

Length of test run was 60 meters and the pressure used was 10 kg.

troduces a variable which must be eliminated in order to secure definitely reproducible data.

To determine whether or not the sand used should be dried, a series of tests was made on cold-rolled steel with the sand as sieved and also as sieved and dried for 24 hours at 105-110 degrees Cent. The results of these tests are shown in Table III.

Excellent checks were obtained with both dried and undried sand. The amount of sand used per test was greater when the dried sand was used, due, no doubt, to the fact that dried sand flows more freely than sand with some contained moisture. Despite the fact that a greater amount of dry sand was used, the loss of weight was greater with the smaller amount of undried sand used in the tests. Since a large volume of sand was sieved and stored and since drying the sand under controlled methods would have slowed up the work considerably, it was decided to make all tests by using the sand without artificial drying.

b. Effect of Pressure and of Distance Travelled by Disk

A series of tests was made on cold-rolled steel to determine the effect of the variables, pressure and distance travelled by the abrading disk. The results of these tests are summarized in Fig. 8. As would be expected, increase in total pressure caused a more rapid loss of weight because of the greater proportional increase in pressure per unit of area, while increase in travel caused a smaller increase in loss of weight due to the decreasing average pressure

per unit of area during the test (greater area of contact). For test purposes it was decided to make all runs at a pressure of 10 kg. and 60-meters travel of the disk.

c. Effect of Carbon Content on the Resistance to Wear

A series of steels varying in carbon content was tested as normalized, as normalized and annealed, and as normalized and hardened, to determine the effect of carbon content on resistance to wear. These results are summarized in Fig. 9 together with the determinations of Rockwell and Brinell hardness. All points on the curves of wear are the average of four tests. The chemical compositions and heat treatments of these steels are given in Table IV.

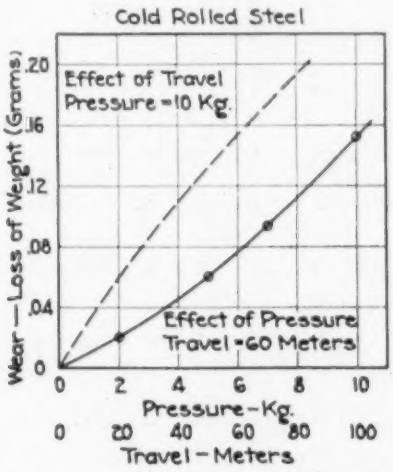


Fig. 8—Effect of Pressure and Distance Travelled by Abrading Disk in Brinell Test.

Table IV
Compositions of the Steels

Mark	Per Cent C	Mn	P	S	Si	Temperature °C. Normalizing	Hardening	Annealing
8D	.078	.482	.099	.083	.018	970	940	920
11D	.203	.430	.006	.041	.027	940	910	890
20C	.425	.673	.044	.026	.223	865	835	815
13C	.573	.700	.013	.023	.200	830	800	780
34A	.762	.501	.028	.026	.276	810	775	755
EU	.81	.23	.024	.021	.28	810	780	755
35A	1.03	.345	.037	.036	.387	810	770	755
51	1.29	.271	.011	.013	.250	925	770	755

All specimens were about ¼-inch thick.
Time of holding at all temperatures, ½ hour.
All hardening by water-quenching.
All specimens hardened or annealed were previously normalized.

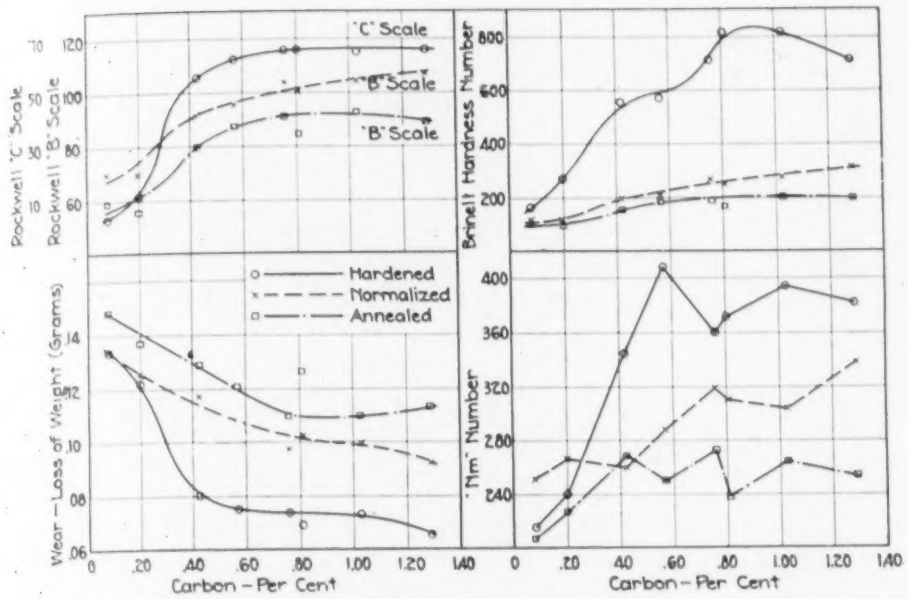


Fig. 9—Effect of Carbon Content on the Wear of Carbon Steels Tested in Brinell Machine.

The "Nm" wear-resistance numbers of these steels were very erratic for reasons previously mentioned and are considered of little value.

If the losses of weight of these steels are considered, it is evident that the percentage of carbon exerted a very marked influence upon the resistance to wear. An increase of carbon increased resistance to wear and this increase was greater in the lower range of carbon content than in the higher range. Furthermore, the resistance to wear of the hardened steels was greater than that of the normalized steels, which, in turn, showed greater resistance than the annealed steels. Comparing these trends with the curves of hardness shows in a convincing manner that the resistance to wear of carbon steels as determined by the Brinell machine is dependent upon the hardness.

The only point which is radically off the weight-loss curves is that for the annealed, 0.81 per cent steel. It will be noted that the hardness of this steel was between the corresponding values for the 0.42 per cent and 0.57 per cent carbon steels and that the loss of weight also was between the corresponding losses of weight of these two steels. Thus, a decreased hardness was reflected by an increased loss of weight. Similarly, the normalized, 0.76 per cent carbon steel showed a slightly higher hardness as judged by

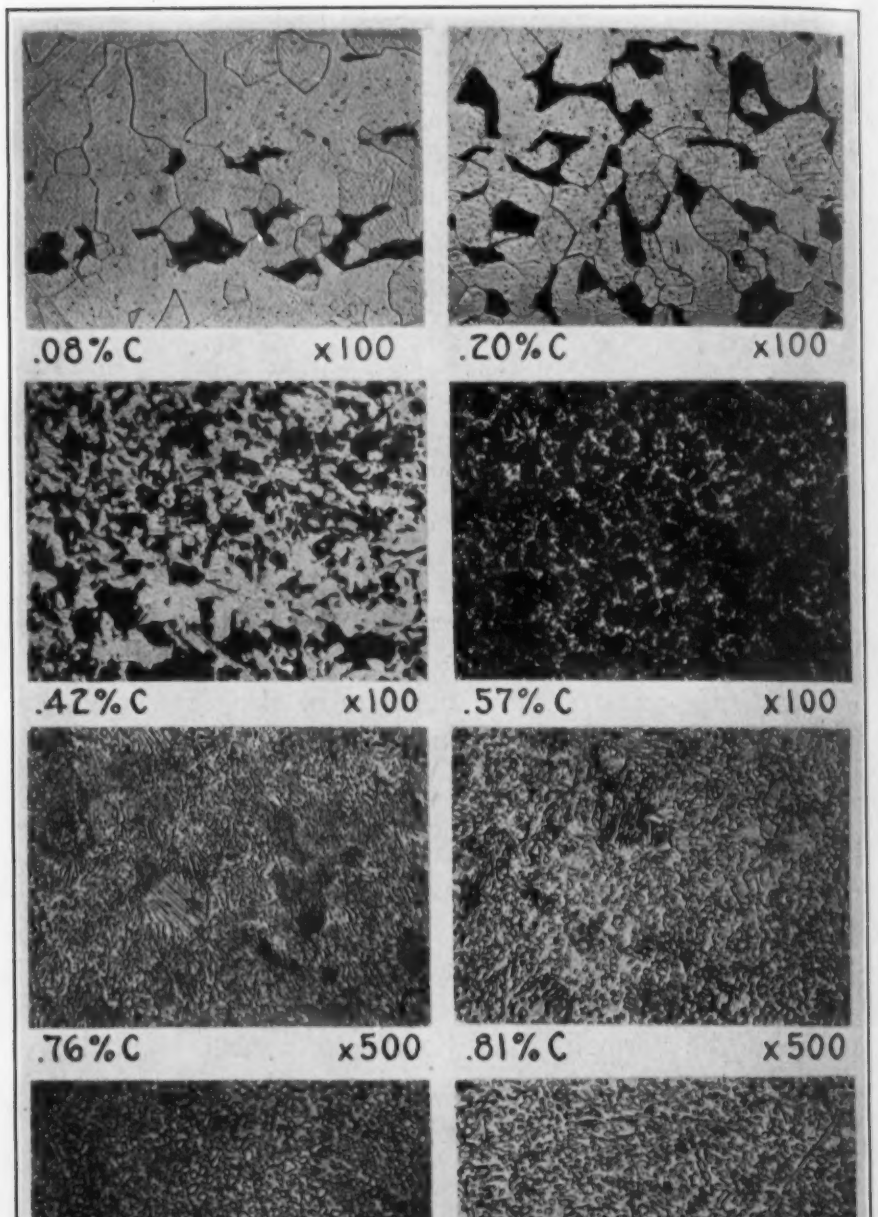


Fig. 10—Microstructures of Annealed Carbon Steels Tested in Brinell Machine. Etched with 5 Per Cent Picric Acid in Alcohol.

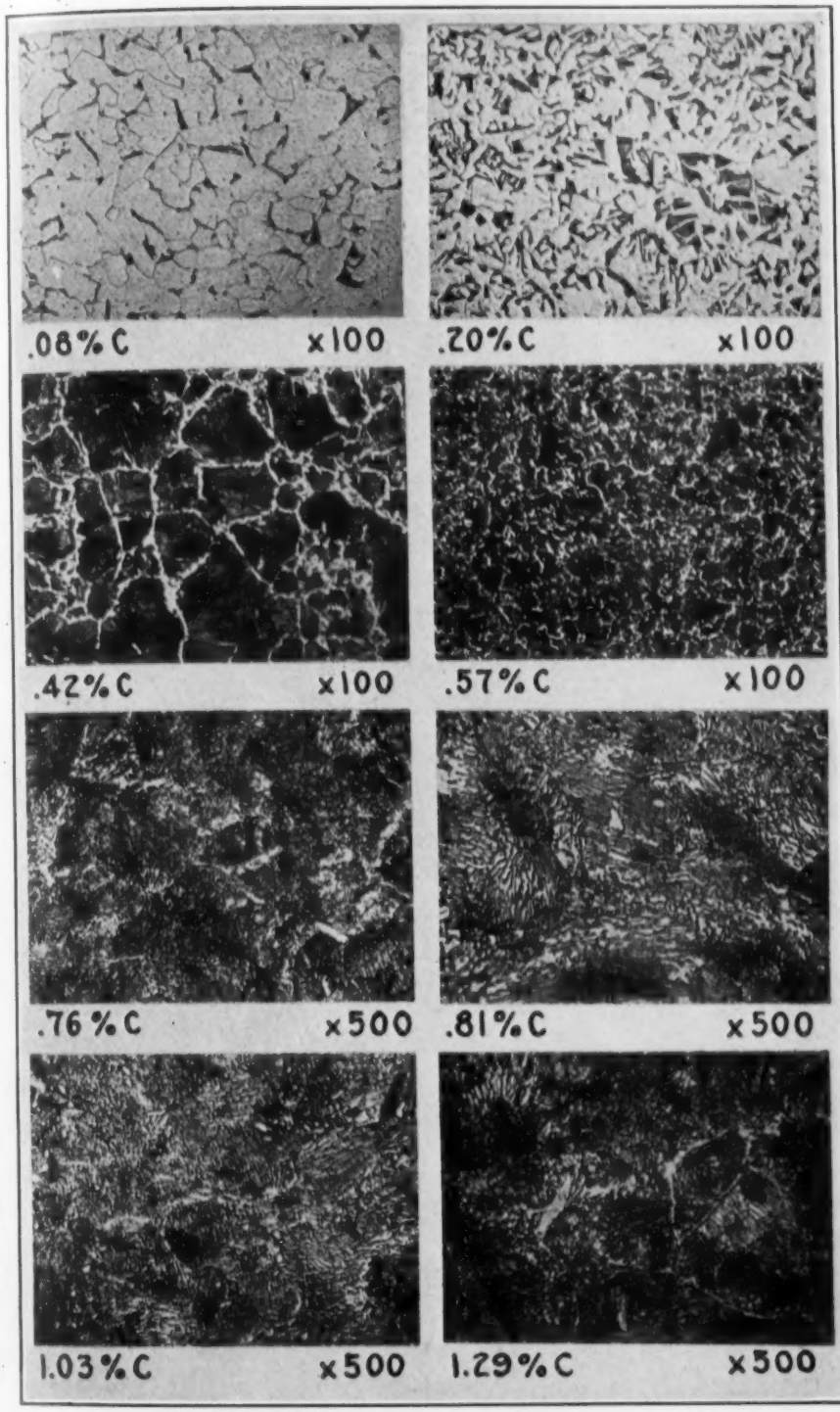


Fig. 11—Microstructures of Normalized Carbon Steels Tested in Brinell Machine. Etched with 5 Per Cent Picric Acid in Alcohol.

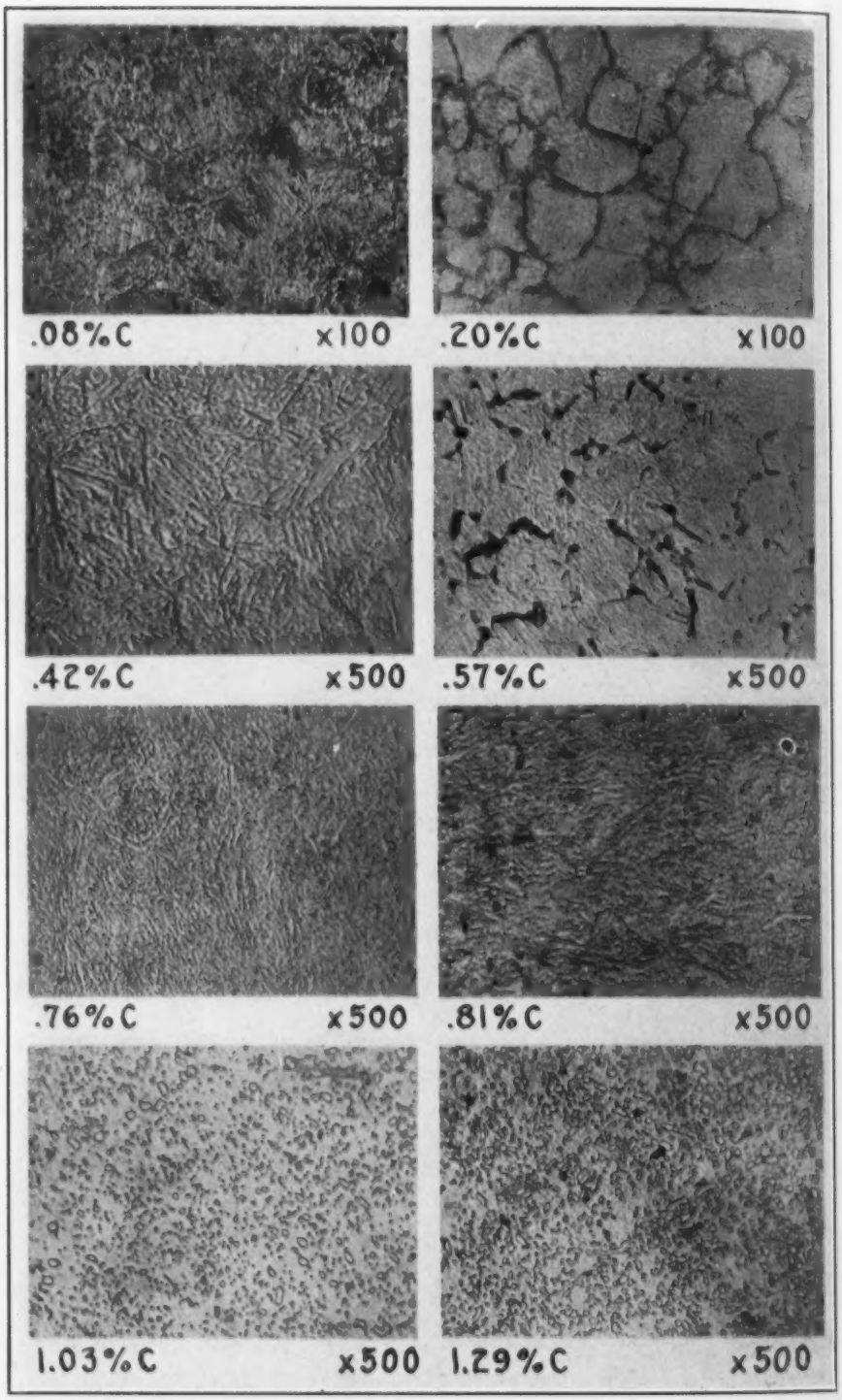


Fig. 12—Microstructures of Hardened Carbon Steels Tested in Brinell Machine. Etched with 5 Per Cent Picric Acid in Alcohol.

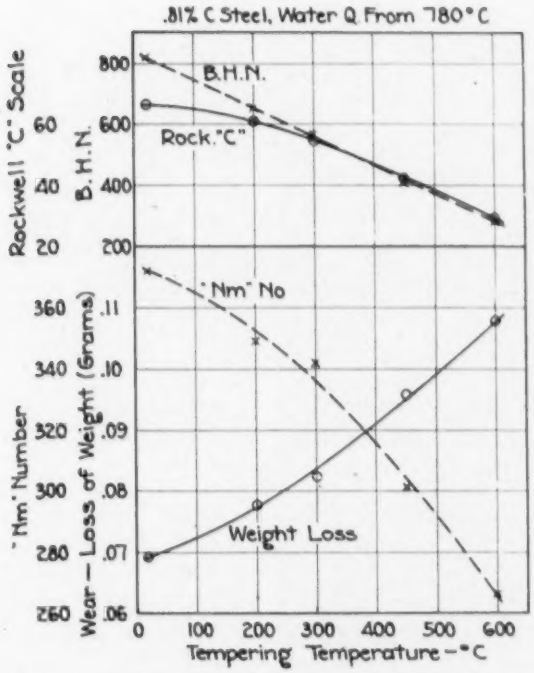


Fig. 13—Effect of Tempering Temperature on the Wear of an 0.81 Per Cent Carbon Steel Tested in Brinell Machine.

the curve of hardness and this was reflected by a decreased loss of weight.

The microstructures of the annealed steels are shown in Fig. 10, of the normalized steels in Fig. 11, and of the hardened steels in Fig. 12.

d. Effect of Tempering Temperature on the Resistance to Wear

The effect of tempering temperature on the resistance to wear of the 0.81 per cent carbon steel was studied. All specimens were held for one hour at tempering temperature. The results of the tests (each point is the average of four tests) together with the hardness determinations are summarized graphically in Fig. 13. These curves show that increase in tempering temperature progressively lowers the resistance to wear of eutectoid carbon steel as determined by the Brinell machine. The microstructures of these steels are shown in Fig. 14.

e. Effect of Annealing Temperature on the Resistance to Wear

There is a popular conception to the effect that the smaller

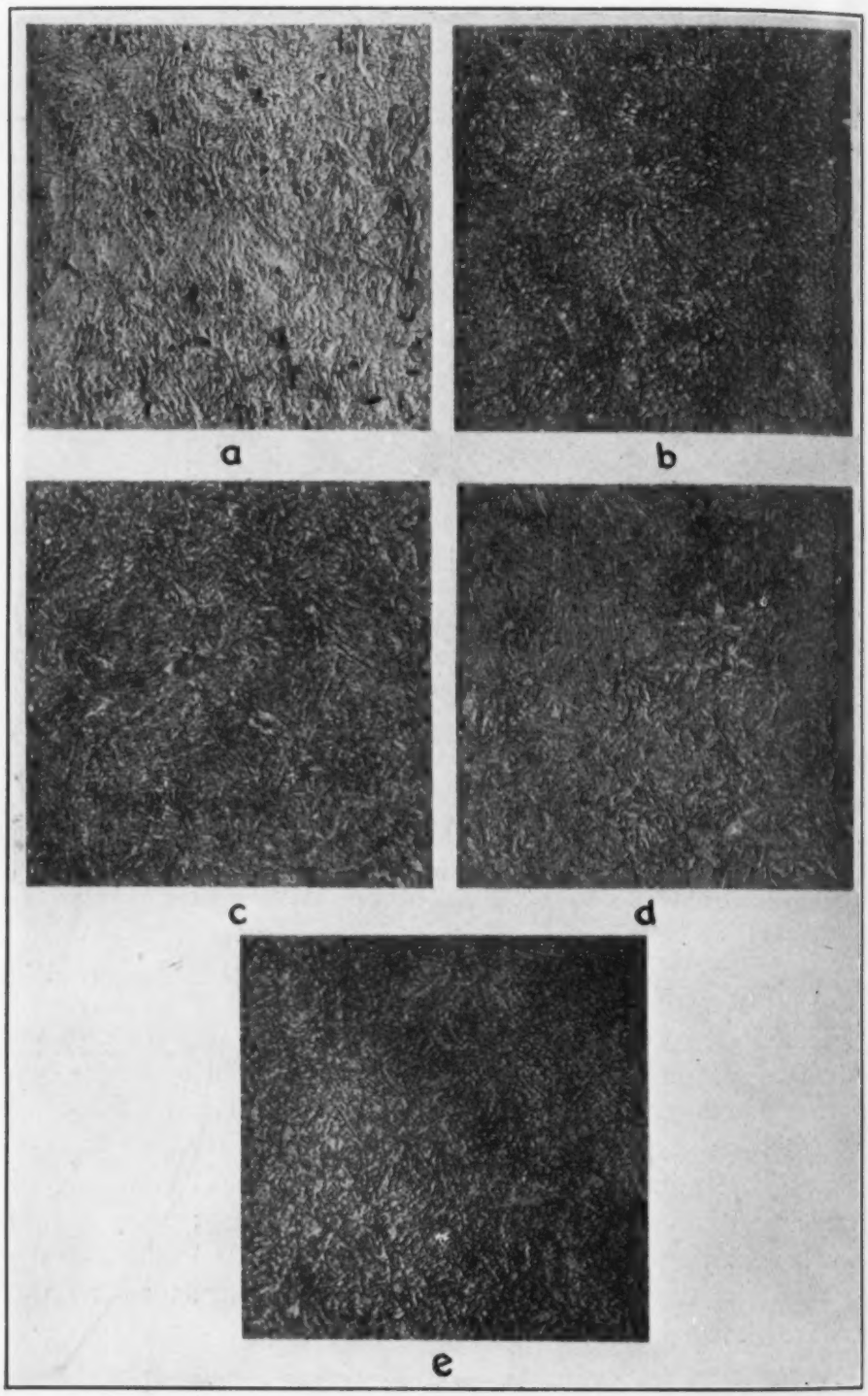


Fig. 14—Microstructures of a Tempered 0.81 Per Cent Carbon Steel Tested in Brinell Machine. Etched with 5 Per Cent Picric Acid in Alcohol. × 500. a—As hardened. b—Tempered at 200°C. c—Tempered at 300°C. d—Tempered at 450°C. e—Tempered at 600°C.

the grain size of the steel, the greater the resistance to wear. This idea seems to be based, at least in part, upon the assumption that the larger the grain size, the more brittle the material and the more easily the individual grains may be torn out. As will be shown later in this report, in the type of abrasion investigated here the individual grains are not torn out as whole grains.

In order to determine the effect of grain size upon the resist-

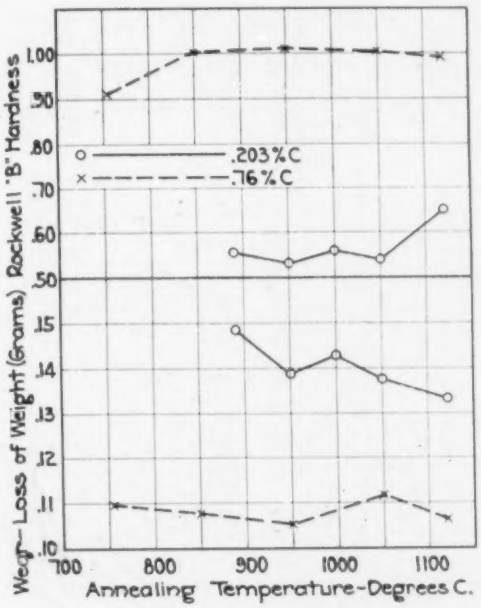


Fig. 15—Effect of Annealing Temperature on the Wear of a 0.20 Per Cent and 0.76 Per Cent Carbon Steel Tested in Brinell Machine.

ance to wear, two steels (0.20 per cent and 0.76 per cent carbon) were annealed at successively higher temperatures and then tested. The samples were held ½ hour at each annealing temperature with the exception of the highest temperature, at which they were held for 3¾ hours. The results of these tests are summarized in Fig. 15.

The effect of the temperature of annealing upon the resistance to wear of the 0.76 per cent carbon steel was very slight, but the effect on the 0.203 per cent carbon steel of increasing the temperature of annealing was rather marked, the resistance to wear increasing with the temperature. This is the reverse of what ordinarily would have been expected and no explanation of this apparent anomaly is offered. It may be noted, however, that increasing the temperature of annealing results in a trend toward higher hardness

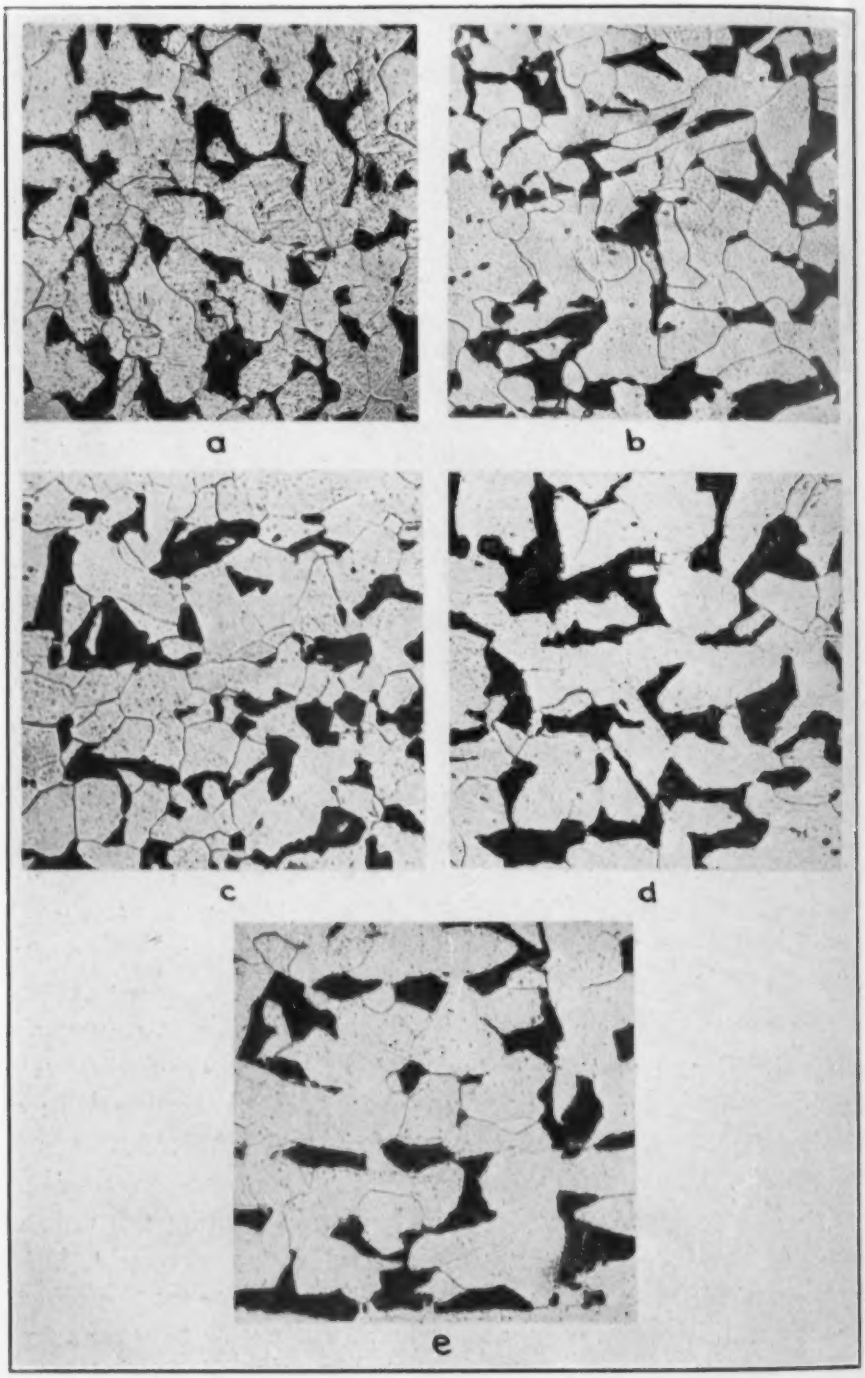


Fig. 16—Microstructures of a 0.20 Per Cent Carbon Steel Annealed at Different Temperatures, Tested in Brinell Machine. Etched with 5 Per Cent Picric Acid in Alcohol. × 100. a—Annealed at 890°C. for ½ hour. b—Annealed at 950°C. for ½ hour. c—Annealed at 1000°C. for ½ hour. d—Annealed at 1050°C. for ½ hour. e—Annealed at 1120°C. for 3¾ hours.

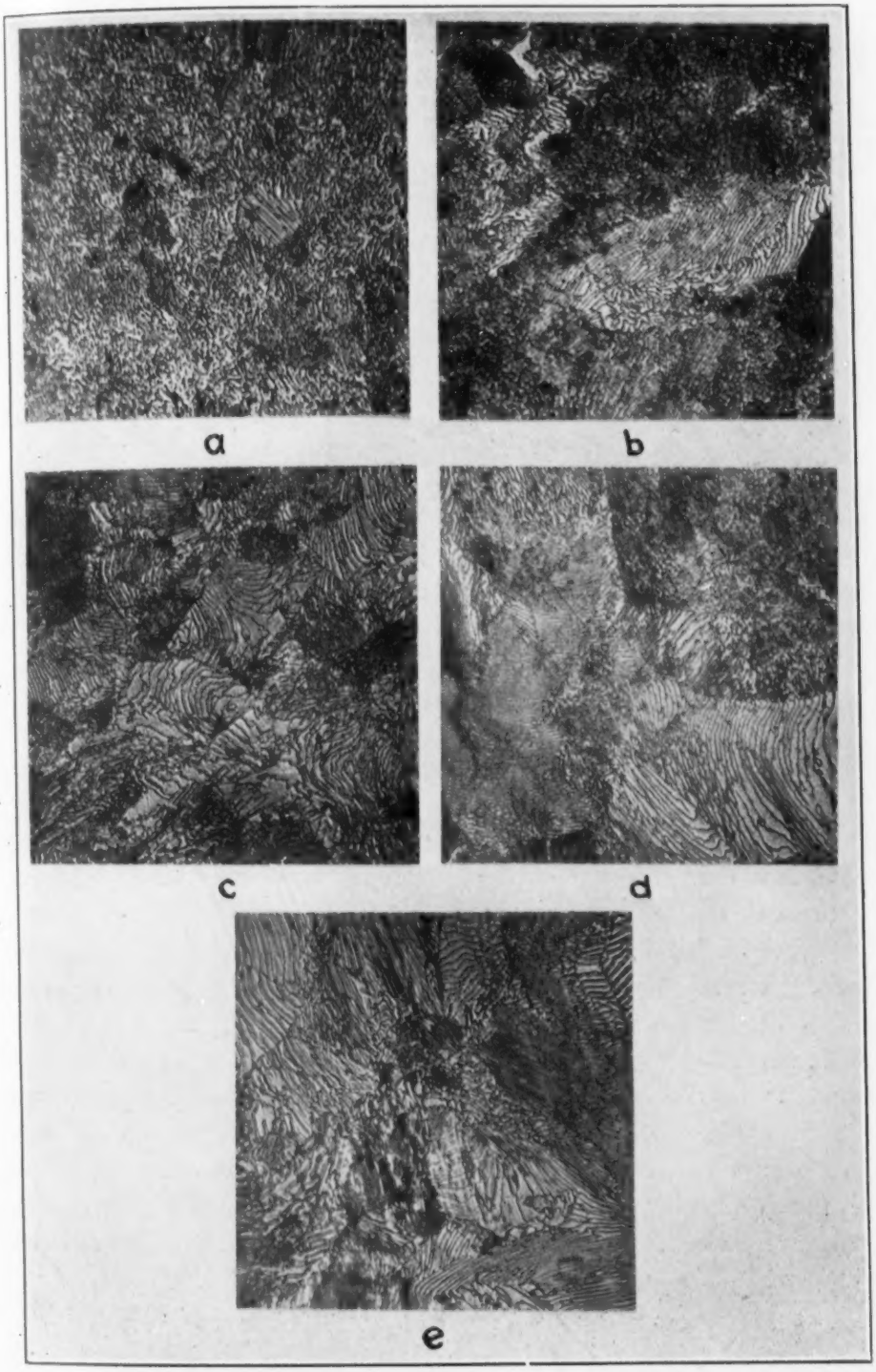


Fig. 17—Microstructures of a 0.76 Per Cent Carbon Steel Annealed at Different Temperatures, Tested in Brinell Machine. Etched with 5 Per Cent Picric Acid in Alcohol. × 500. a—Annealed at 755°C. for ½ Hour. b—Annealed at 850°C. for ½ Hour. c—Annealed at 950°C. for ½ Hour. d—Annealed at 1050°C. for ½ Hour. e—Annealed at 1120°C. for 3¼ Hours.

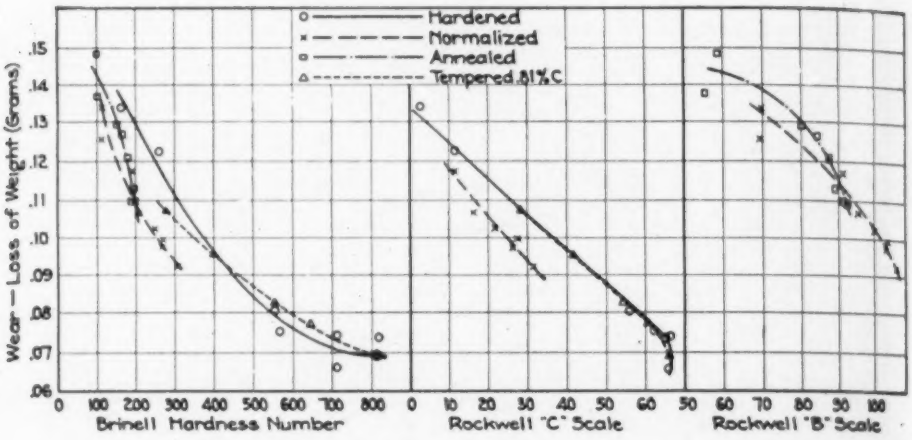


Fig. 18—Effect of Hardness on the Wear of Carbon Steels as Determined by the Brinell Machine.

and this is a factor in causing increased resistance to wear. The microstructure of the 0.203 per cent carbon steel, annealed at various temperatures, are shown in Fig. 16, and of the 0.76 per cent carbon steel in Fig. 17. The increase in grain size is not so marked as might have been expected, but the trend toward larger grains with increasing temperature is unmistakable.

f. Relation of Hardness to Resistance to Wear

The fact that the wear as determined by the Brinell machine is influenced by the hardness has already been mentioned. In Fig. 18 the data shown in Figs. 9 and 13 have been replotted to show the effect of hardness on the loss of weight of the various carbon steels. These curves show that for increased hardness, the losses of weight decrease; that is, the resistance to wear increases. This relationship is practically linear in the lower range of hardness numbers; in the higher range, however, apparently large changes in hardness are not accompanied by correspondingly great changes in resistance to wear. This may be due to the fact that the Brinell hardnesses of the hard steels were found to be unduly high, caused perhaps by the difficulty of reading accurately small Brinell impressions.

g. Examination of Abraded Surfaces

In order to show the manner in which wear takes place on abraded surfaces, the groove worn into an annealed 0.20 per cent

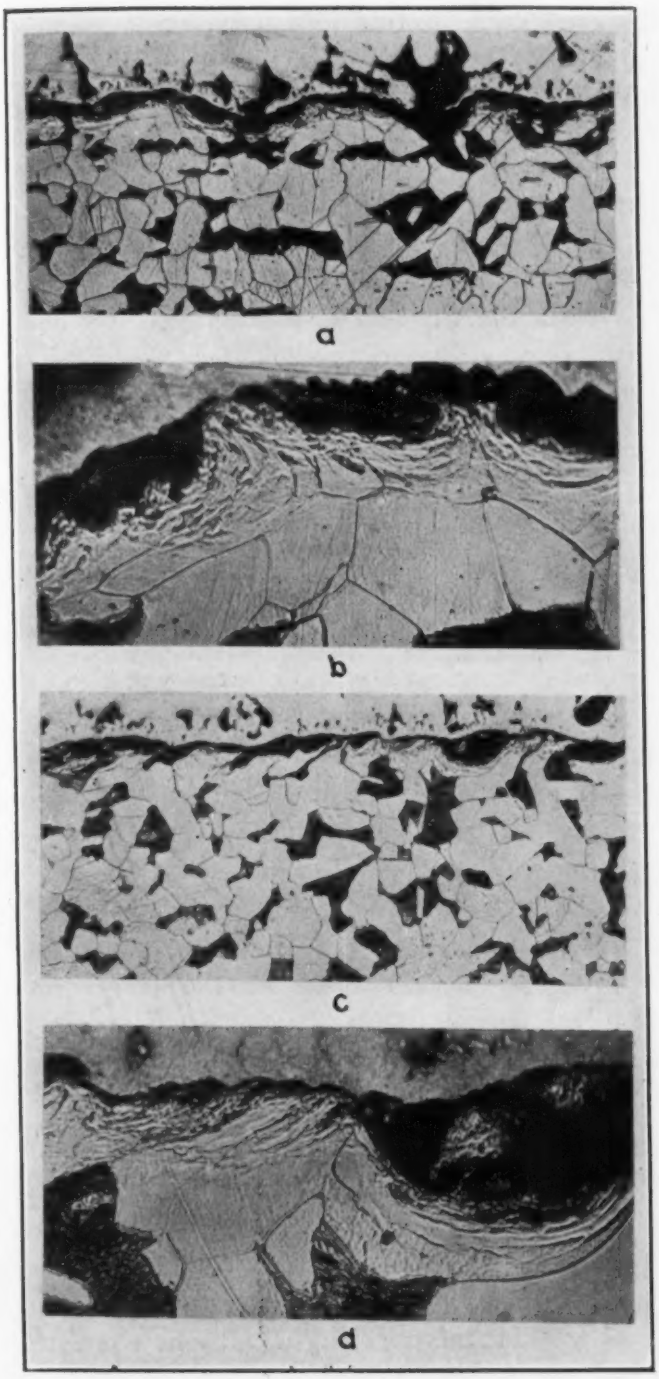


Fig. 19—Micrographs of the Structure Adjacent to the Abraded Surface of an Annealed 0.20 Per Cent Carbon Steel Tested in Brinell Machine. Etched with 2 Per Cent Nitric Acid in Alcohol. a—Cross section through bottom of groove. × 100. b—Same—× 500. c—Longitudinal section through bottom of groove. × 100. d—Same—× 500.

carbon steel was copper-plated and both cross sections and longitudinal sections were cut for microscopic examination. The structures of these samples at the wearing surfaces are shown in Fig. 19.

The appearance of the surface both at right angles to the groove and longitudinally through it is about the same. In both cases very marked evidence of localized flow in the form of strain is present. This has also been found in wear-resistance tests of bronzes.[7] An examination of the abraded surface (see, for example, Fig. 19b) also reveals the fact that the mechanism of wear takes place by parts of individual grains being abraded and not by grains being removed in their entirety. This is what would be expected when it is considered that at room temperatures the cohesive force between the grains is greater than the internal strength of the grains.

V. Discussion of Results

1. Ball-mill Test

The results obtained with the ball-mill were quite erratic and will be dismissed as of qualitative value only. The ball-mill does not seem to hold much promise as a laboratory machine for testing the resistance of metals to abrasion. The trouble with this machine is two-fold—it is not sufficiently sensitive to show positive differences in resistance to wear and it is not sufficiently accurate to give check results. In addition to these disadvantages the making of the spherical specimens is a costly and tedious process.

These tests showed, however, that the forged and hardened specimens lost less weight than the normalized specimens.

2. Sand-blast Test

The sand-blast is a very rapid and accurate abrading medium. Of all the steels tested, however, no great differences were apparent between them. Practically the same rates of wear were obtained, for instance, on the same steel when normalized as were obtained when hardened. It is thought that this apparent lack of sensitivity was due to the fact that the abrading conditions were not sufficiently severe to bring out the inherent differences in wear characteristic of the materials tested. This opinion is based upon the fact that

[7] H. J. French, S. J. Rosenberg, W. LeC. Harbaugh and H. C. Cross, "Wear and Mechanical Properties of Railroad Bearing Bronzes at Different Temperatures," B. S. Journal of Research, I (R. P. 13); September, 1928.

differences in resistance to wear between some materials were apparent at a blasting pressure of 60 pounds per square inch that were not noticeable at a pressure of 30 pounds per square inch.

3. *Brinell Test*

A series of carbon steels varying from 0.08 to 1.29 per cent carbon was tested in the normalized, annealed, and hardened conditions. The results of these tests showed that the resistance to wear of these steels in this special type of test was dependent upon the hardness. A study of the effect of tempering temperature upon the wear showed the same trend, i. e., the resistance to wear increased with the hardness.

The effect of grain size upon the resistance of wear was studied by testing a 0.20 per cent and a 0.76 per cent carbon steel annealed at various temperatures. An increase in temperature of annealing resulted in a general increase in resistance to wear as evidenced by a decreased loss of weight, this being more marked in the low-carbon steel than in the high-carbon steel.

A study of the mechanism of wear or abrasion showed that grains of the steel were not torn off in their entirety but were worn away in small particles, the abrasion being accompanied by severe local straining.

VI. Summary and Conclusions

Three devices for testing the resistance of metals to abrasion were studied—the ball-mill, the sand-blast, and the Brinell machine.

The ball-mill proved to be very unsatisfactory, only qualitative results being obtained. Generally speaking, this type of test showed hardened steels to be somewhat more resistant to abrasion than the same steels in the normalized condition.

The sand-blast proved to be a very rapid and accurate abrading medium but failed to differentiate between many steels which were known to have widely different wear characteristics in different conditions of service. This apparent anomaly seems to be due to the fact that the abrasion was not sufficiently severe to bring out the differences in resistance to wear inherent in the steel and shows conclusively that slight modifications of test conditions will give different results on the resistance to wear of different materials.

The Brinell test showed that the resistance to wear of normalized, annealed, hardened, or tempered carbon steels increased with

an increase in hardness. Increasing the temperature of annealing resulted in a slight increase in resistance to wear, the increase being more marked in a low-carbon than in a high-carbon steel.

Abrasion in this last type of test is caused by the wearing away of exposed sections of the individual grains and not by the removal of the grains in their entirety. The abrasion is accompanied by severe local straining due to cold working.

As slight variables in testing for resistance to abrasion have been shown to give different results, it should not be expected that wear-resistance tests made on machines of this type will necessarily check special service conditions where many variables are encountered.

ACKNOWLEGMENTS

The author is indebted to Dr. R. L. Dowdell, senior metallurgist, for his kindly suggestions and criticisms, and to Houston Babb, junior scientific aid, for his conscientious assistance in many of the tests.

DISCUSSION

Written Discussion: By Albert G. Zima, development and research department, International Nickel Co., New York City.

The problem of producing steels to withstand abrasion is often a perplexing one indeed. It seems that each application must first require a certain amount of experimental work before one is sure of the performance he may expect of the steel in question.

Wear caused by abrasives on metal may be roughly classified into two types. Under one type we may consider the severe abrading action of coarse rock or similar substance under increased pressure and magnitude of impact. The second type includes the action of such materials as sand, coke, slag, cinders, grain, etc., passing over a metal under various conditions of velocity and pressure. It is this latter type with which we are concerned in the discussion of Mr. Rosenberg's paper.

The results of the sand blast tests under the 30-pound blast, shows no difference in wear resistance between the 12-per cent manganese steel, the two chromium-molybdenum steels and white iron. Mr. Rosenberg attributes this equality to the lack of severity of the abrading conditions. This theory, however, is not entirely in accordance with results obtained in actual field tests. For example, it has been demonstrated time and again that in conveyors or chutes handling coke, cinders, slag, etc., hardened alloy steels or white iron outwear 12-per cent manganese steel by a wide margin. However, as the conditions of abrasion become more severe due to pressure, the manganese steel begins to assert itself.

A few years ago the writer had occasion to experiment with some small

impeller cups. These cups were attached to arms approximately 8 inches in length and rotated about an axis at approximately 1800 revolutions per minute. The rotating cups encountered a stream of coke breeze and hurled it outward with considerable force. The average life of the 12-per cent manganese cups was 2 hours. Several experimental cups were cast from a heat of chromium-nickel-molybedum steel and subsequently heat treated. It was found that the cup life increased in proportion to the hardness, the best results obtained being 8 hours. Surface defects such as blow holes or cold shuts invariably caused premature wear. A machine of this type might serve well as a wear testing apparatus since it is not difficult to maintain uniform conditions of operation.

In conclusion, there is no doubt that Mr. Rosenberg's excellent paper will be read with considerable interest and further contributions on the subject of abrasion will be well received by makers of steel.

Author's Closure

Some few years ago Dr. Gillett, in discussing some results of wear tests presented before the American Society for Testing Materials, remarked that sometimes investigators know the answers to certain questions before they start to work, and that if their theories fail to check the known facts, it is their theories which are wrong, and not the facts. This comment seems pertinent to the case in hand.

It is well known that 12-per cent manganese steel shows its high resistance to abrasion only when used under conditions which cause the steel to work harden. Thus when used in a steam shovel to excavate rocky ground, it appears to last wonderfully well, but when used to dig into soft sand or clay, it seems to be of little use. Thus, it is seen that any one wear test may or may not show the superiority of 12-per cent manganese steel over, say, a chromium-molybdenum steel, depending upon whether that especial test does or does not produce severe work hardening of the material tested.

In the sand-blast test described, severe work hardening did not take place. Some work hardening did occur, else the manganese steel would not have shown up as well as it did. This test, then, is not a fair test of 12-per cent manganese steel.

In the tests described by Mr. Zima, the conditions were not such as to produce work hardening of the materials used. It is doubtless because of this fact that the manganese steel showed to such poor advantage when compared with the chromium-nickel-molybdenum steel.

CORROSION TEST FOR RESEARCH AND INSPECTION OF ALLOYS

By William R. Huey

Abstract

In this paper the need for an accurate corrosion test is emphasized. The relation is indicated between corrosion resistance of alloys and structural condition as influenced by heat treatment. The trend of development toward greater improvement, especially in the acid resistant alloys seems to point to a slight variation in chemical composition and closer control of heat treating temperatures. In order to evaluate the effects of minor changes such as these, it is necessary to use a corrosion test capable of producing consistently accurate data. A test designed for this purpose and for the purpose of inspection of alloys is described. Data obtained from the described test are given to illustrate the accuracy possible. These data happen to be taken from a number of test results obtained during the study of the effect of acid temperature on corrosion resistance of the so-called stainless alloys.

THE acid resistant alloys of chromium, nickel and iron have rapidly created and filled an important role in industry. Chemical processes, not possible a few years ago, are today in operation, because the former need for equipment has been met. The story of this phenomenal advance in alloy development is too well known to warrant further repetition. It is not, however, so well known that this group of alloys, under certain conditions, will exhibit an erratic tendency, particularly in corrosion behavior.

Chrome-iron and chromium-nickel-iron alloys respond so sharply to treatment by heat, that close control must be exercised of the various heating operations necessary to fabrication. The American Society for Steel Treating is interested in heat treatment of steel to produce desirable mechanical properties not present in the untreated metal. For centuries, the primary purpose for heat treating has been to alter the mechanical performance of metal. During

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, a member of the society, is engaged in process research work for the E. I. DuPont De Nemours and Co., Wilmington, Delaware. Manuscript received June 23, 1930.

recent years, however, another objective has become evident, which is involved in altering the chemical performance. That this evidence has been noted, is attested to by the present existence of patents, claiming as new this feature of the ancient art. There is here a new phase of heat treating to claim the attention of the Society.

Possible Trend of Alloy Development

Corrosion resistance and other properties of a metal are dependent upon the heat history, that begins the moment the ingredients are charged into the melting furnace. By heat treatment, tacitly assumed proper, the alteration of metal performance, whether mechanical or chemical, is toward improvement; conversely, mistreatment by heat, causes inferior performance. At some point along the route from raw material to finished article, the service behavior of the alloy is determined definitely and often irrevocably. Skill in the manipulation of temperature as a weapon against corrosion has not yet reached the high degree of perfection that will be achieved in the future. A more thorough knowledge of the effect on metal of temperature, confined to very narrow limits, will ultimately result in even greater improvement of corrosion resistance by existing alloys than that made during the past decade.

Much of the experimental investigation that has led to the discovery of the present-day acid resistant and high strength alloys, has been based on the mixing of metals and concerned with methods to foster the mixing, to produce the alloys. Some work has been done to determine the effect of slight, closely controlled variations of the minor elements, or impurities, in the alloys; but this phase of alloy research has been handicapped by the lack of sufficiently exacting test methods for measuring the effect. The mathematically possible combinations of major elements in alloys have been fairly well developed. The line of future development lies in the small variation of the minor, or heretofore rarely used elements.

The trend of this development is evident in patents recently issued covering the addition of small amounts of tungsten and limiting of carbon in chromium-nickel alloys. This is but a mere beginning of the list of similar discoveries that are yet to be made. When a method is available for minutely measuring the changes in properties caused by such slight variations in chemical composition, it will be found that the effect produced is disproportionately great,

when only the more obvious features of the cause are considered. For instance, sulphur and phosphorus are considered incidental impurities in steel and other ferrous alloys. Might the future not reveal that slight variation in these elements is enormously valuable in determining the properties?

Present Need for Accurate Corrosion Test

If it is fairly obvious that the trend of alloy development is toward slight variation in heat treatment and close manipulation of apparently minor elements, then it is certainly obvious that today, more than ever before, a corrosion test to produce consistently accurate data is of vital importance. An exacting test of this nature is also important to the user of alloys, for practical economic reasons, to determine whether the alloys have been given the proper care in the various steps of the manufacturing process. The user of the chromium-nickel-iron alloys needs such a test, especially. If a single test could be devised to embody the two-fold purpose of research and inspection, it would be of equally great value to metal producer and metal user.

The interest in corrosion is as widespread as the ravages of the process, and co-existent with this interest, to a large degree, is the interest in corrosion testing. The technical literature bears ample witness to the prevailing interest in both the subjects; and the vast amount of experimental investigation reported shows both to be involved one in the other. There are a great many manifestations and phases of corrosion, and, likewise, almost as many types of corrosion tests, designed usually to serve some special purpose. Many tests have some outstanding characteristic worthy of commendation, a few have none, and very few may be said to serve all purposes. Many fall short of the intended purpose by the sheer weight of the complex technique necessary for successful execution.

The author does not intend to present an abstract, critical or otherwise, of the technical literature pertaining to the subject of corrosion testing. This has been ably done by the makers of the literature. Those interested in the subject will find the contributions of Evans, Pilling, Ackerman, Fraser, Bengough, Stuart and Lee, Friend, Schulz, Vernon, McKay, Thompson, Saklatwalla and many others, replete with reference to, and acknowledgment of, the work done by each other. With due acknowledgment of

the inspiration and valuable aid to be found in the contributions by these investigators, a humble attempt is made in this paper to:

1. Point out the vital need for an accurate quantitative corrosion test, to be used for research investigation and inspection of alloys.

2. Describe a quantitative test capable of producing highly accurate data.

Broadly speaking, attention to the subject of corrosion may be directed along three different but closely related lines of investigation, e. g.:

1. A study of the fundamental aspects of corrosion, designed to arrive at some concept of the mechanism of the process. Such a study encompasses a wide range of chemical, physical and metallurgical principles, and a thorough knowledge of the relations between them. It is doubtful that a true understanding of the nature of corrosion, in the general sense, can result from the work of an individual. The combined endeavor of a group of workers, each the master of one of the branches of human knowledge mentioned above, will be necessary.

2. The investigation of the reaction of metals to corrosion for the specific purpose of improving the corrosion resistance of existing metals, or for development of new metals. This more specialized phase of the subject holds promise of immediate material return of value to industry.

3. The development of a method for predicting the performance of a metal which is to be used under corrosive conditions. This line of investigation is distinct from that mentioned under (2) only in purpose.

The lines of investigation indicated above each require a method for measuring the attack of corrosive materials on metals. Especially in the execution of (2) and (3), the method required must be simple and capable of producing exacting, accurate data that may be reproduced at will.

Description of Corrosion Test

An experimental investigation of the corrosion resistance of chromium-iron alloys, carried out almost continuously since 1924 in the laboratories of the E. I. du Pont de Nemours Company has developed a corrosion test that fully meets the requirements of (2)

and (3) above. The preliminary phase of the experimental program consisted of laboratory and plant testing of a large number of alloys of this type. This work revealed the need for very accurate control of the testing conditions; the second part of the program was, therefore concerned exclusively with the development of a more depend-

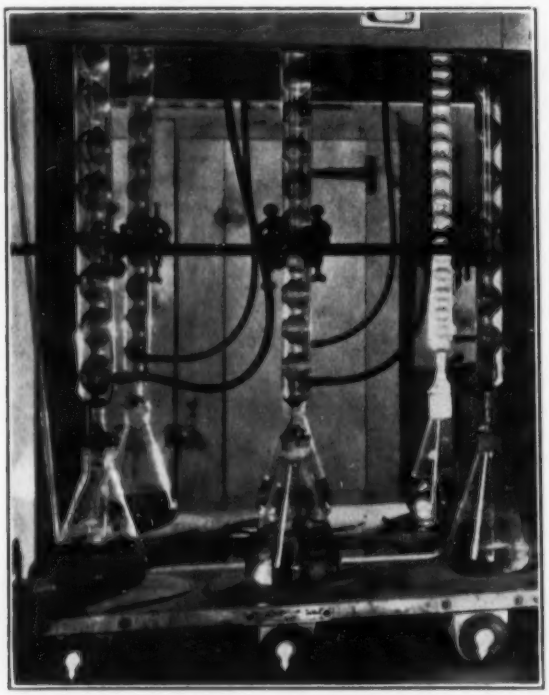


Fig. 1—Photograph of Testing Apparatus.

able laboratory test than was at first available. Thousands of hours of laboratory testing since 1927, devoted to the study of factors influencing the corrosion resistance of chromium-iron alloys, have yielded data confirming the opinion that a highly accurate, quantitative, test has been developed.

In this test, carefully prepared samples of the metal are fully immersed for five 48-hour periods in nitric acid (65% HNO_3 by weight) contained in all-glass apparatus, and maintained constantly at the boiling point. The outstanding characteristic of the test is the simplicity of method by which the many variable factors are eliminated that are known to affect the reproducibility and accuracy of various test results. It has been demonstrated by the consistent accuracy of many determinations, and the detailed description to follow will indicate, that the chief variable factors taken into account

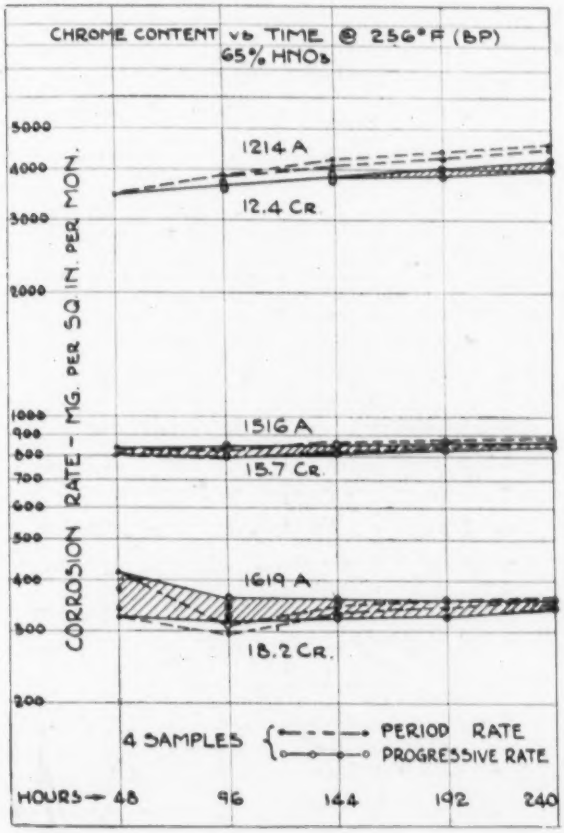


Fig. 2—Corrosion Rate of Four Samples Each of Three Different Alloys. Chromium Content Range of 6.40 Per Cent.

are temperature, acid strength, oxygen content of corroding solution, liquid film on sample surface formed during tests and preparation of sample.

Temperature—By using an acid having approximately the concentration of constant boiling solution, (68.5%) the variation in temperature never exceeds three degrees Fahr. If occasional observation shows ebullition taking place, it may be safely assumed that the temperature is within 3 degrees Fahr. of the boiling point of 65% HNO_3.

Acid Concentration—There is no change in acid strength during the period of the test, providing sufficient cooling water is passing through the reflux condenser on the flask. The constant boiling concentration of the acid is further insurance against change in strength. A number of analyses of acid have been made which have never shown variation exceeding 0.01 per cent. Sufficient volume of

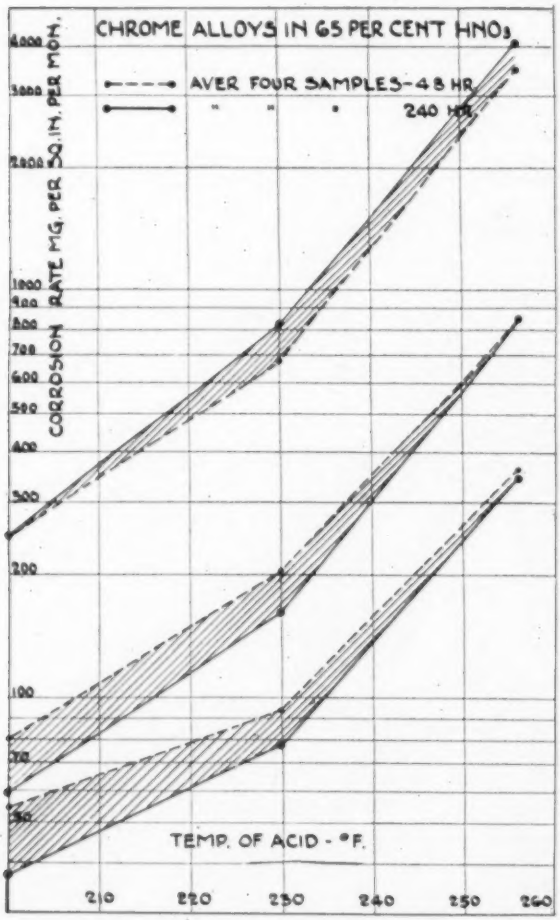


Fig. 3—Effect of Acid Temperature and Alloy Composition on Corrosion Resistance.
Upper Curve 12.4% Chromium
Middle Curve 15.7% Chromium
Lower Curve 18.2% Chromium

acid is used so that change in strength is negligible, due to chemical reaction with the metal. The corrosive action of 65 per cent nitric acid on the chromium-iron alloys is sufficiently severe to cause a measurable rate of a magnitude that sharply reveals significant differences in the resistance of different alloys of the group. The presence in the acid of a very small amount of impurities, such as hydrochloric, sulphuric and hydrofluoric acids, cause discrepancies that are disproportionately great. For this reason, pure nitric acid must be used.

Oxygen Concentration—The necessity for providing a known supply of oxygen to the solution is avoided by eliminating oxygen in the boiling. This troublesome source of discrepancy in corrosion

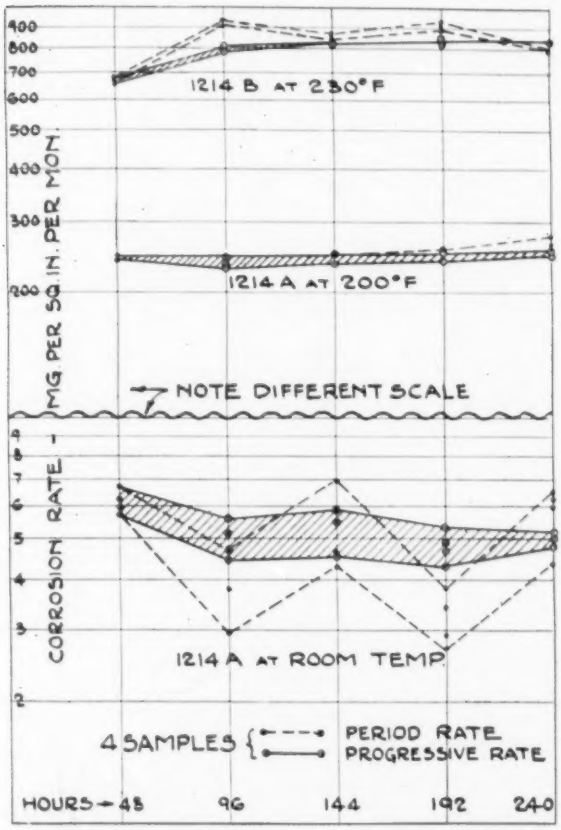


Fig. 4—Variation in Corrosion Rate Between Four Samples of 12.4 Chromium Alloy.

test results can be avoided only with difficulty when it is attempted to control oxygen concentration by the uncertain means of aeration.

Liquid Film—Under certain conditions, it has been reported by Brunner, there is a tendency for formation of a thin film of liquid on the surface of the specimen which may be a source of discrepancy in results obtained. The consistent accuracy of the data obtained with this test indicate that the constant movement of liquid at the metal surface caused by boiling must diminish the effects of the liquid film.

Sample Surface and Size—Influencing variation in the surface of the sample has been avoided by careful preparation of a surface free from visible scratches or tool marks, but *not* polished to mirror-like finish. Care is necessary in selection of abrasive paper similar to 120 French emery, renewed frequently to prevent contamination of sample surface with scale particles. Heating of the sample by

too rapid abrasive action must be avoided. The size of samples must be in proportion to the volume of solution used, and convenient for weighing on a finely adjusted laboratory balance.

The testing apparatus is shown in Fig. 1. It consists of a flask, fitted with a reflux condenser, integral with a ground glass stopper. In the wall of the stopper are glass hooks for hanging the glass rods supporting the specimen in the solution. The heat is supplied by a three-burner electric hot-plate, having "low," "medium" and "high" adjustment. Auxiliary equipment consists of grinding and polishing wheels, for sample surface preparation, and a laboratory balance capable of weighing accurately to tenths of a milligram. For testing at temperature below the boiling temperature of acids, it is, of course, necessary to use a thermostatically controlled bath of water or oil.

It has been found convenient to use metal samples approximately 1.875 by 0.875 by 0.375 inch with 0.25 inch diameter hole in the center of one end about 0.25 inch from the edge. If, it is preferred, however, there is no objection to the use of a one-inch diameter by 0.375 inch disk. After machining the surface, the tool marks are removed with 120 French emery, taking care to renew the paper frequently and to avoid too rapid abrasive action causing heat. The emery treatment is continued until visible scratches have been removed; but a mirror-like surface is not desirable.

After surface preparation, the sample is carefully cleaned, especially of grease or oil. Exact measurment is made for computing the total area and density is determined by water displacement, if it is not known by previous determination. After thorough cleaning and drying, the weight is determined to an accuracy of one-tenth milligram.

The acid should be made to 65 per cent by weight HNO_3 from commercially pure acid and analyzed for impurities. About 750 cubic centimeters of the acid are placed in the flask and the weighed specimens immersed to a depth that prevents contact with the bottom of the flask and at a distance from each other that prevents contact with each other or the sides of the flask. After placing the reflux condenser and starting the cooling water through it, the hot-plate switch is set to "high." When the acid begins boiling, the switch is set at a point where boiling will be maintained. It is possible to test three samples in one flask, but different metals should

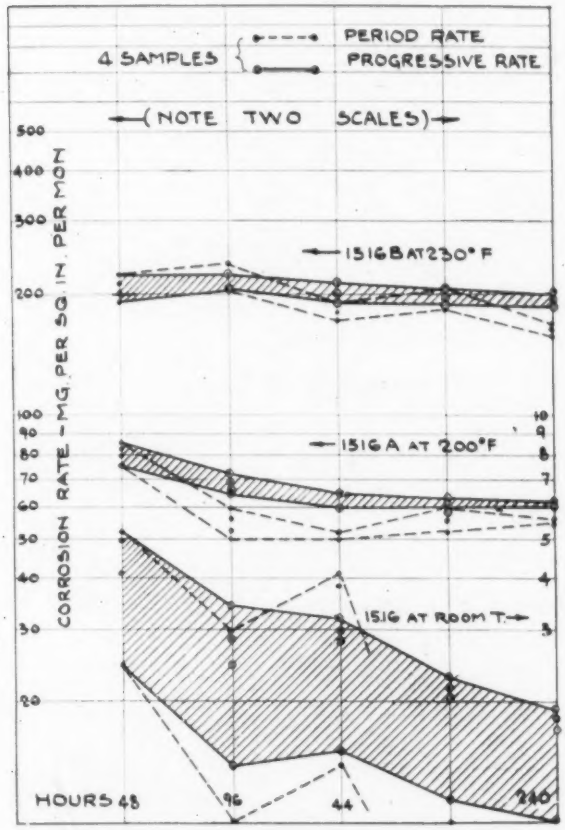


Fig. 5—Variation in Corrosion Rate Between Four Samples of 15.8 Chromium Alloy.

not be tested together. Occasionally a piece of wet litmus paper placed over the exit of the reflux condenser will detect whether there is sufficient water passing through to completely condense the vapors from the boiling acid. After exactly 48 hours have elapsed the samples are removed, quickly washed in water, thoroughly dried and weighed. The chromium-iron and chromium-nickel-iron alloys should be tested for five 48-hour periods, which is ample time for a constant rate to develop. Each period the acid should be renewed to avoid any possibility of contamination by corrosion products affecting the results. It is important to maintain boiling constantly and throughout the entire 48-hour period.

The corrosion rate for each 48-hour period and progressively for the entire 240 hours can be calculated from the determined data as inches penetration per month, which is one method of expressing results, taking into account the density of the metal. The formula

Table I
Alloys Tested

Alloy Designation	Chemical Composition Per Cent Cr	C	Mn	P	S	Si	Rockwell B Range		Tested at Degrees Fahr.
1214A	12.38	.09	.35	.010	.019	.24	70	78.5	Room; 256
1214B	12.35	.08	.40	.014	.023	.28	72.5	76	200; 230
1516A	15.72	.08	.30	.012	.021	.15	75	81	Room; 256
1516B	15.98	.09	.35	.010	.021	.28	78	78.5	200; 230
1619A	18.20	.09	.38	.013	.016	.24	79	84.5	Room; 256
1619B	18.75	.07	.37	.013	.023	.26	79	81.5	200; 230

used for calculating this value is $R = \frac{43.9\ W}{A\ S\ T}$ where W is weight loss in grams; A is area in square inches; S is density; T is time in hours; and 43.9 is a factor converting cubic units to linear units and hours to month.

There is considerable objection to expressing corrosion data as inches penetration per month. The psychological reaction to such a value is to immediately interpret the laboratory test data in terms of service life of equipment in the process. Those not familiar with the fallacy of such a precedure will quite naturally assume that a process vessel made of half-inch plates of a metal showing 0.01 inch per month penetration rate in laboratory test, will be corroded completely in four years and two months, by the acid used in the laboratory test. If corrosion and corrosion testing were so straightforward and simple in nature there would not be such a vast literature concerning the subjects. No method of expressing corrosion test results developed to date can be relied upon to truly represent the case, so that laboratory tests may be used to predict process performance. Visual observation of the tested specimens is always a valuable aid in rating the specimens in relative order of merit.

The test method described in the foregoing paragraphs yields accurate quantitative data that may be used in research to show the effect on corrosion resistance of slight variations in heat treatment or chemical composition, or in determining whether a given alloy is normal with respect to corrosion resistance. *The corrosion rate obtained shows the resistance of the sample tested to the corroding solution used under the conditions of the test made.*

Acid Temperature Against Corrosion Rate

Using this test, and modification of it, the author is attempting

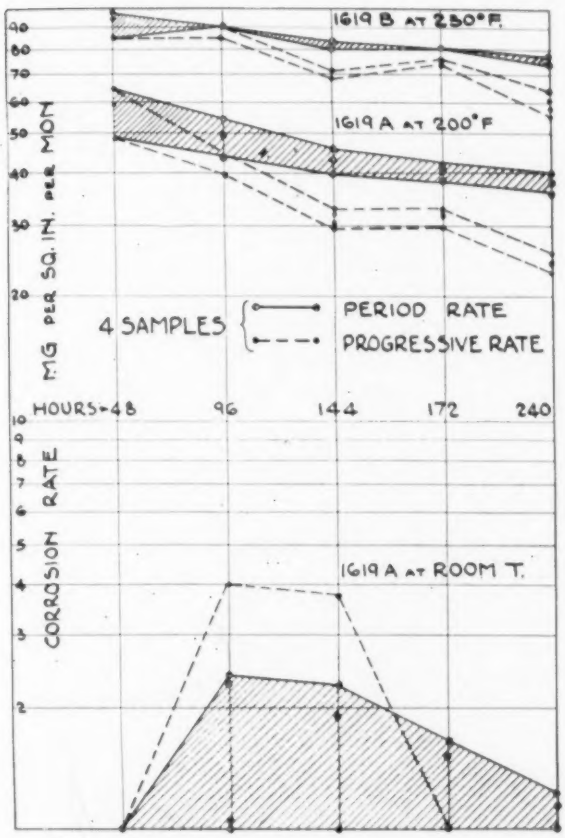


Fig. 6—Variation in Corrosion Rate Between Four Samples of 18.2 Chromium Alloy.

to attain one of the chief objectives of corrosion testing research; namely, to establish a relation between carefully controlled laboratory test conditions and process performance of metals. As a part of the experimental program planned for this purpose, certain data were obtained to show the relation between acid temperature and resistance. In collecting these data, the standard test procedure described in foregoing paragraphs has been altered somewhat, but in spite of the alteration, it is believed this small part of a general research program will serve as a representative example of the accuracy of the method.

Table I shows the chemical composition, and hardness after test, of the alloys tested. The corrosion rate for the various temperatures was determined over a period of two years whenever the work could be scheduled in the general experimental program. Therefore, the alloys used were of slightly different chemical composition, but the difference in composition is probably not sufficiently great to obscure

Table II
Example of Data Obtained—240 Hrs. in 65 Per Cent HNO_3 Boiling

Alloy	Area in Square Inches (1)	(2)	(3)	(4)	Weight in Grams (a) Before Test (b) After Test (1)	(2)	(3)	(4)	Corrosion Rate (a) Inches Per Mon. (b) Mg. Per Sq. In. Per Mon. (1)	(2)	(3)	(4)
1214A	5.53	5.54	5.53	5.49	74.8025	75.0873	74.5098	74.1874	.03183	.03214	.03133	.03146
					67.3666	67.5650	67.1904	66.8872	4055	4100	3990	3980
1516A	5.51	5.46	5.49	5.48	73.6128	73.7310	74.3354	74.2250	.00663	.00663	.00671	.00675
					72.0719	72.2026	72.7805	72.6628	839	834	849	852
1619B	5.45	5.51	5.51	5.48	74.7238	73.5628	73.7692	73.5726	.00281	.00272	.00264	.00268
					74.0774	72.9304	73.1571	72.9545	356	345	334	339

Purity of Alloy 1214A—7.73
Purity of Alloy 1516A—7.72
Purity of Alloy 1619B—7.69

the major trend with respect to the effect of acid temperature on the corrosion resistance. In making these experiments, the standard test procedure described herein was altered only in the use of a carefully regulated constant temperature bath of paraffin in place of the electric hot-plate used in the boiling test.

In the curves of Fig. 2 are shown the corrosion rates of four samples, each of three different alloys representing a chromium content range of 6.40 per cent. While the extreme difference in rate between low and high chromium alloys is in this case 3650 milligrams per square inch per month, the greatest variation in rate for a given alloy during a given test interval is 90 milligrams per square inch per month. This occurred during the first period with the 18.2 chromium alloy which showed a mean rate of 370 milligrams per square inch per month. The variation is approximately 24 per cent of the mean rate. Note that this alloy showed variation of only 10 milligrams per square inch per month at the conclusion of the test, or 3.1 per cent deviation, based on the minimum rate at the beginning of the test. During this period the variation in weight loss for the four samples was 1.4 milligrams, while the weight of samples ranged between 72930.4 milligrams and 74077.4 milligrams. Therefore the measurable loss was approximately 0.002 per cent.

Fig. 3 shows the relation between acid temperature and corrosion resistance of the three alloys. It is obvious from these data that the same relation between temperature and resistance holds for each alloy. The greatest variation occurs at temperatures below boiling, which is to be expected on account of the effect of dissolved oxygen and quiescent acid. However, the variation is between the

average of four samples for 48 hours test and the average of four samples for 240 hours test. The data provide ample testimony in favor of testing at boiling point and for the longer period test.

Figs. 4, 5 and 6 show in every case the wide variation in rate between four samples of each alloy at room temperature. It is to be noted that the variation decreases as the temperature increases and as the length of test increases. The value is demonstrated of the long test period and the boiling temperature in producing closely agreeing data.

It should be noted that the test data reported herein are incomplete with respect to showing the effect of acid temperature on resistance. Further tests are in progress in the range between room temperature and 200 degrees Fahr. where it is suspected, a critical temperature exists, at which the rate increase changes noticeably. The partial data presented in the curves show rather conclusively, however, the high degree of accuracy possible by using the test.

The corrosion test described will yield *accurate quantitative data that represent truly the corrosion resistance of the metal sample in the test acid used.* Interpretation of the data depends upon the knowledge of the metal tested and somewhat upon the knowledge of the acid properties. If erratic results on several samples of the same metal are obtained, it may be safely assumed that these samples have inherently erratic properties.

Acknowledgment

The author wishes to express appreciation for the permission to publish this paper, given by the E. I. du Pont de Nemours Company. Credit is due F. G. Thedick and G. A. Sands for assistance in execution of the experimental program.

DISCUSSION

Written Discussion: By Dr. F. N. Speller, director of research, National Tube Co., Pittsburgh.

The author has shown how the chemical attack in nitric acid may be measured with consistent precision by controlling the various factors affecting the corrosion rate but he says nothing about the possible effect of such chemical action on the physical properties. It is often important to determine, by physical test (such as tensile and impact) on the metal before and after chemical tests, whether the metal has suffered any serious deterioration either due to intergranular corrosion or to changes in the

structure, due to prolonged heating or both. Other reagents probably have more effect in this respect but it would be interesting to know whether the author has made such tests. Permanence of physical properties is often of more importance than resistance to chemical attack.

Bengough, Stuart and Lee[1] report their results in water and dilute salt solutions to be reproducible within ± 1%. This and the work of Mr. Huey and others indicate that corrosion laboratory experiments may now be carried out within the same range of accuracy as many other experiments in physical chemistry.

Written Discussion: By C. L. Hippensteel, Bell Telephone Research Laboratories, New York City.

The author has developed an ingenious test which gives unusually closely agreeing results which are highly desirable and not usually attained in the case of most of the corrosion testing procedures which are being used at the present time. The author has wisely stated, we feel, that the information to be obtained in his test is related only to the particular type of acid being used.

He points out that his test is capable of detecting the effect on corrosion resistance of the alloys of slight heat treating temperature changes and of minor impurities. We feel that the value of the paper would be enhanced had the author presented data demonstrating these points.

Written Discussion: By R. J. McKay, superintendent of technical service, International Nickel Co., New York City.

Mr. Huey has described a test which is very useful for the inspection of alloys and for other purposes. In this test almost the complete range of nickel-chromium-iron and chromium-iron alloys is corroded resulting in a clearer picture of the metallurgical features affecting corrosion than from tests in which a large majority of samples must be reported as unaffected. His results should be accepted as particularly conclusive since they come from the laboratories of the DuPont Co. where there is opportunity to study metals of widely varying manufacture under widely varying corrosive conditions. Mr. Huey and the DuPont Co. are to be thanked for giving us their frank and unbiased opinion on this subject.

As a proponent of a different type of corrosion testing apparatus, which has been thoroughly described in papers by the writer and others, I would like to state that, according to the standards used in designing this other apparatus, Mr. Huey's apparatus can be relied on to produce true results. Indeed he has proven this by the results quoted and by his frank statements as to agreement or disagreement of these results with each other and with service observations. This type of corrosion testing and clear readable presentation cannot but be useful to metallurgists interested in establishing the facts on these alloys. This is particularly true since the knowledge obtained by laboratory tests is being run a close race if not outdistanced by the current rapid commercial adoption of these alloys.

Regarding questions on which I might wish to suggest modifications of

[1]Bengough, G. D., Stuart, J. M., and Lee, A. R., "The Theory of Metallic Corrosion in the Light of Quantitative Measurements—Part III," Proc. Royal Society, A, Vol. 127, pp. 42-70, 1930.

Dr. Huey's findings, there are certain details which might be usefully considered. Mr. Huey has rendered a service in enumerating the points which must be controlled to make a dependable corrosion test. I have also attempted in a former paper to enumerate such conditions and would add to this list, with which I am in full agreement, two points which he has controlled (but not mentioned) namely, galvanic or concentration cell effects which are eliminated by the method of holding the samples and second, rate of movement of corroding agent past the sample which in this case is controlled by the boiling and discussed by Mr. Huey under the head of liquid film. Regarding sample surface, this is quite largely a question of solid films and might also be discussed under this heading. Regarding aeration, Dr. Huey's statement on pages 1132 and 1133 is rather a direct refutation of some of the writer's own statements. However, any disagreement is largely a surface one as there certainly can be no disagreement as to the importance of controlling aeration. In practical work the amount of aeration is probably the variable which most affects the corrosion rates. Lack of consideration of it was a factor which made corrosion rates under apparently similar conditions mysteriously different until the situation was clarified. Extremely useful results in corrosion testing are to be obtained with properly designed aerating apparatus as well as by the complete air removal which Mr. Huey has used.

As a somewhat different phase of this same important subject, namely the effect of oxidizing or reducing conditions on resistance of metals to corrosion, I shall give the following figures which may help in determining where Mr. Huey's valuable test should be used. The figures are taken from Technical Publication No. 174 of the A. I. M. E. "Resistance of Iron-nickel-chromium Alloys to Corrosion by Acids" by Pilling and Ackerman. Reading from Mr. Pilling's curves on page 13 and 9 of this paper covering rates of corrosion in sulphuric acid and in nitric acid the following relations are brought out. In 5 per cent sulphuric acid, with the 20 per cent chromium 10 per cent nickel and balance iron alloy, the rate of corrosion is about 5000 mg./dm^2/da.; with 15 per cent nickel this rate is reduced 800 mg./dm^2/da. a change of over 5 to 1 with the addition of nickel reducing corrosion rate in that ratio.

On the other hand, taking the corrosion rates of alloys in 5 per cent nitric acid, the 20 per cent chromium 10 per cent nickel balance iron alloy gives a rate of about 1000 mg./dm^2/da. while the 15 per cent nickel alloy has a rate almost exactly the same. In other words, in this particular alloy range the addition of nickel does not affect the corrosion rate in nitric acid. These tests were made in an apparatus which will compare with Dr. Huey's in reproducibility of results. Thus Dr. Huey's test, if it is to be applied to corrosion under non-oxidizing conditions, will give incorrect results unless a less oxidizing solution than nitric acid is used. It is under these non-oxidizing conditions where the corrosion rates of the chromium bearing alloys are highest, that nickel additions produce their greatest effect. This advantageous effect of nickel has also been noted in the wide range of service conditions where the 18-8 alloys have been introduced. Pos-

sibly the nickel bearing alloys are primarily used because of their valuable physical and fabricating qualities but they also produce desirable additional resistance to rusting. The test which Dr. Huey has described requires modification to indicate this proven value of a considerable nickel content.

Author's Closure

The author is indebted to Messrs. C. L. Hippensteel and F. N. Speller who have kindly submitted written discussions on the paper of the above title.

These discussions clearly show the necessity for a statement placing emphasis on the *single* purpose of the paper. It is intended that the paper point out the necessity for an accurate corrosion test for the research investigation and inspection of present day corrosive resistant alloys. A test for this purpose is briefly described and typical data obtained during the successful use by the author of the test are presented.

The author had hoped that the italics on pages 1136 and 1139 of the preprint would serve the usual purpose in emphasizing the most important statements made in the paper, namely:

1. "The corrosion rate obtained shows the resistance of the sample tested to the corroding solution used under the conditions of the test made."

2. "The corrosion test described will yield accurate quantitative data that represent truly the corrosion resistance of the metal sample in the test acid used."

It is unfortunate that the abstract contains the sentence: "The relation is indicated between corrosion resistance of alloys and structural condition as influenced by heat treatment." No specific data are given in the test to support the statement. A happier choice of words would have been "implied" rather than "indicated." The author did not feel that data demonstrating the effect of heat treatment and minor impurities could have been appropriately placed in the paper. It is hoped that these data may be treated in a separate paper.

In his discussion, F. N. Speller has raised the question of the effect on physical properties of metals of the test attack. In cases where permanence of physical properties is of sufficient importance and where the corrosive attack causes severe alteration of physical properties, this effect can be readily determined by using appropriate test specimens. A complete test should include, of course, full examination of specimens before and after being subjected to the action of the test, but it has been found, by the author and others that weight loss provides a rather reliable indication of the effect of the test on other properties of the metal. The author favors an occasional checking of the relation between weight loss and loss in tensile properties, as well as the frequent use of the microscope in determining the type of attack under various conditions.

In discussing a subject of such broad complexity as that chosen by the author, it is impractical to attempt to include all the possible phases of the subject. In purposely limiting the scope of the paper, the author has undoubtedly omitted many important points that would be of interest and that

would possibly have an appropriate place in the discussion. It is hoped that further papers by the author and others may present separate phases of the general subject in the detail they deserve.

In preparing the plates for printing, an omission was made in Fig. 3, of the preprint. The three sets of plotted data should be labeled 12.4 chromium, 15.7 chromium and 18.2 chromium, beginning at the top of the plate.

In his discussion, Mr. McKay has raised the question which requires further comment than that given in the paper. Mr. McKay has pointed out conclusively the difference in action of oxidizing and reducing acids on alloys, particularly of the high chromium-nickel type. The author acknowledges this limitation of the test as described in his paper. The test as described has been applied to other metals than the so called stainless alloys where it was necessary to use acids other than nitric. The test procedure is not restricted to the use of nitric acid. Any acid or liquid may be used in this test, providing there is no decomposition caused by heating and providing the liquid has a low enough vapor tension so that the condenser can prevent change in concentration due to evaporation. The author has used in other experiments than those described, sulphuric acid, acetic acid, mixed acids of nitric and sulphuric, hydrochloric acid and several other solutions of various chemical compounds. Some mention of this point should have been made in the paper but many such points could not be discussed fully in the interest of coherence and brevity.

Mr. McKay reports a difference in the effect of 5 per cent sulphuric acid and 5 per cent nitric acid on chromium-nickel alloy containing 10 per cent nickel in one instance and 15 per cent nickel in another instance. The nitric acid did not reveal any marked difference in the corrosion rate for the increase of 5 per cent nickel while the sulphuric acid did show a decrease of about 5 to 1 in the rate favoring the higher nickel alloy.

Experience with the test described, using 65 per cent nitric acid at the boiling point and the 18 chrome 8 nickel type of alloy compels the remark that this test would show the appropriate difference in the two alloys. The difference would probably not be so great as that reported by Mr. McKay for the 5 per cent sulphuric acid.

Probably the difference in opinion here is due to the use of two totally different tests. The author believes that the difference in attack of the two acids on the two alloys described by Mr. McKay can be more clearly demonstrated using the aerated test and the weaker acids. However, for the purpose of merely demonstrating the effect of increased nickel content or any other alloy variable, it is believed that the test as described would be adequate. On the other hand, for predicting what might happen in process to equipment made of samples tested in the laboratory, the question raised by Mr. McKay certainly deserves consideration, providing the investigator is fully aware of the danger involved in attempting to predict plant equipment performance strictly on the basis of laboratory tests.

COPPER BRAZING IN HYDROGEN ELECTRIC FURNACES

By H. M. Webber

Abstract

This paper discusses the copper brazing process utilizing electric furnaces with hydrogen atmospheres, as developed and applied by the laboratory and factory staffs of General Electric Company. An explanation is given for the ready flow of molten copper into tight steel joints, it being stated that results with the process improve as contact pressures increase. Considerable strength is obtained, as well as gas tightness, because of: (1) the formation of metallic solutions, (2) the penetration of copper along grain boundaries of the iron, and (3) the tendency for grain growth across the joints.

The discussion touches upon effects of the relatively high furnace temperatures required, as well as effects of the hydrogen atmosphere upon steel. Some applications of the process are cited, followed by references to furnace design and apparatus for producing the gases required for furnace atmospheres.

COPPER brazing in electric furnaces with properly controlled atmospheres has been practiced for several years, but comparatively little has been written on the technical aspects of the subject. The fundamental process is a rather old one, but its value in application was not realized until a copper braze was accidentally made in 1906 during experiments on the reduction of tungsten oxide. The oxide was being heated on a copper boat in an iron tube using a furnace with hydrogen atmosphere. As the reduction progressed the temperature got beyond the operator's control, melting the copper boat. Upon cooling, it was found that the tungsten metal was solidly brazed to the iron tube. Two years later, in 1908, the first practical application was made of this "discovery," tungsten contacts being copper brazed to steel parts in hydrogen electric furnaces, for use on automobile induction coils. From that date General Electric Company has built up within its own organization a great variety of applications, has developed electric furnaces

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, H. M. Webber, is associated with the industrial heating and welding engineering department, General Electric Co., Schenectady, N. Y. Manuscript received May 29, 1930.

using hydrogen atmospheres, and made other improvements to a point where the process can now be proclaimed valuable to the manufacturing industry as a whole.

The process differs from the well known forms of brazing with brass or copper by the torch flame, blow pipe or dip methods, in that the pieces to be brazed are brought up uniformly to temperature in an electric furnace, without flux, in a protective and reducing atmosphere. Hydrogen serves the double purpose of flux by cleaning the surfaces, as well as excluding air. Copper is placed on the parts near the joints to be brazed, in any convenient form such as wire, chips, powder, paste, etc. By this process all the joints in an article can be brazed in one passage through a furnace, and uniformly good results are obtainable, with most of the "personal factor" of the other methods eliminated. In one instance, five pieces each containing over 14,000 joints, were brazed in individual passages through a furnace, and during a subsequent test under 150-pound air pressure not a single one of the joints leaked.

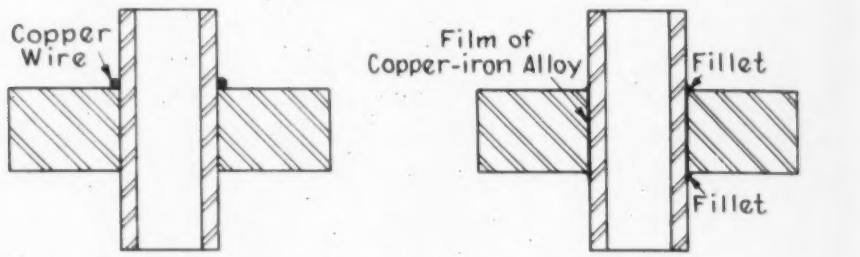
An illustration of how the copper braze functions is shown in Fig. 1. "A" indicates a piece of clean steel tubing pressed into a clean hole in a steel disk. Around the joint at the top is placed a ring of copper wire, completing the preparation for copper brazing. The assembly is placed in a furnace having a hydrogen atmosphere, at temperature above the melting point of copper about 1150 degrees Cent. (2100 degrees Fahr.), and as the copper melts it flows into the joint, alloying with the steel and forming fillets at the upper and lower extremities of the joint, shown at "B." The joint will be gas-tight and strong. If such parts are properly assembled and brazed, upon twisting or pulling the steel will frequently fail before the brazed joint will yield.

The ready flow of copper into tight joints is due to the attraction of iron and copper for one another, and to the capillary force available. These effects can be best illustrated by common examples.

When a piece of clean glass is dipped into water the liquid rises in a curve against the glass, because the surface of the glass has a slightly greater attraction for molecules of water at the surface, than the cohesive attraction which water molecules at the surface have for one another. The surface tends to enlarge until the two forces come to equilibrium. However, should the clean piece of glass be dipped into kerosene oil, the oil would gradually

spread up over the entire surface of the glass, because the attraction of the glass for kerosene exceeds by a considerable amount the surface tension of the oil. This effect has been frequently observed in the common appearance of oil films on the outside surfaces of kerosene lamps.

Molten copper wets and flows upon the surface of clean hot iron much faster than kerosene spreads over glass, because of the ability of copper to alloy with the iron. As copper penetrates into the surface of iron, the rate of inward diffusion decreases rapidly,



A. Object prepared for copper brazing. Ring of copper wire adjacent to clean, tight joint.

B. Same object as shown in "A". Copper brazed in hydrogen electric furnace. Temperature 2100°F. (1150°C.). Copper flowed into joint by capillary attraction, forming metallic solutions.

Fig. 1—An Illustration of How the Copper Braze Functions. (Left) Object Prepared for Copper Brazing. Ring of Copper Wire Adjacent to Clean, Tight Joint. (Right) Same Object. Copper Brazed in Hydrogen Electric Furnace. Temperature 1150 Degrees Cent. (2100 Degrees Fahr.). Copper Flowed into Joint by Capillary Attraction, Forming Metallic Solutions.

and because of the relatively great attraction of adjacent areas for copper, the copper creeps along the iron surface. Copper brazed objects often indicate that copper flows upwards on the surfaces just as it flows downwards. Equilibrium curves show 8 to 10 per cent copper in solution with iron at 1150 degrees Cent., with about 2 per cent remaining in solution upon cooling.

Photomicrograph Fig. 2 shows the edge of a low carbon steel rod which was immersed in a crucible of molten copper, and allowed to soak 19 hours at 1200 degrees Cent. (2190 degrees Fahr.) in hydrogen, at the end of which time the mass was cooled and a section taken. A considerable amount of iron went into solution with the copper during the soaking period, as evidenced by the fact that the iron rod diameter decreased from 0.500 inch to 0.465-0.44 inch. The sectional view illustrates the mechanism by which copper has penetrated the iron. It seems to dissolve the iron or iron-rich phase



Fig. 2—Showing the Edge of a Low Carbon Steel Rod which was Immersed in a Crucible of Molten Copper, and Allowed to Soak 19 Hours at 1200 Degrees Cent. (2190 Degrees Fahr.) in Hydrogen, at the End of which Time the Mass was Cooled and a Section Taken. The Sectional View Illustrates the Mechanism by which the Copper has Penetrated the Iron.

by solution at the grain boundaries. The dark constituent outlining the iron sample plainly marks the area of diffusion of copper into the iron. Globules of the iron-rich phase are seen floating off into saturated copper, the copper-rich phase consisting of about 3 per cent iron and 97 per cent copper after cooling.

The illustration described above is an exaggeration of the effects produced in a copper braze, because of the long soaking period involved, but it does point out the manner in which the metallic solutions are formed, and aids in visualizing the ability of the copper to make a strong bond with steel.

When a glass capillary tube is lowered into water, the water climbs up inside the tube because of the combined action of surface tension and the attraction of glass for the water. The tendency to climb, or the upward force of the water surface, is inversely proportional to the radius of the tube. Therefore, in a small capillary tube the water will climb faster and farther than in a large capillary tube, because the upward force is greater in proportion to the downward pull of the column.

Molten copper is much more fluid than water, and is drawn

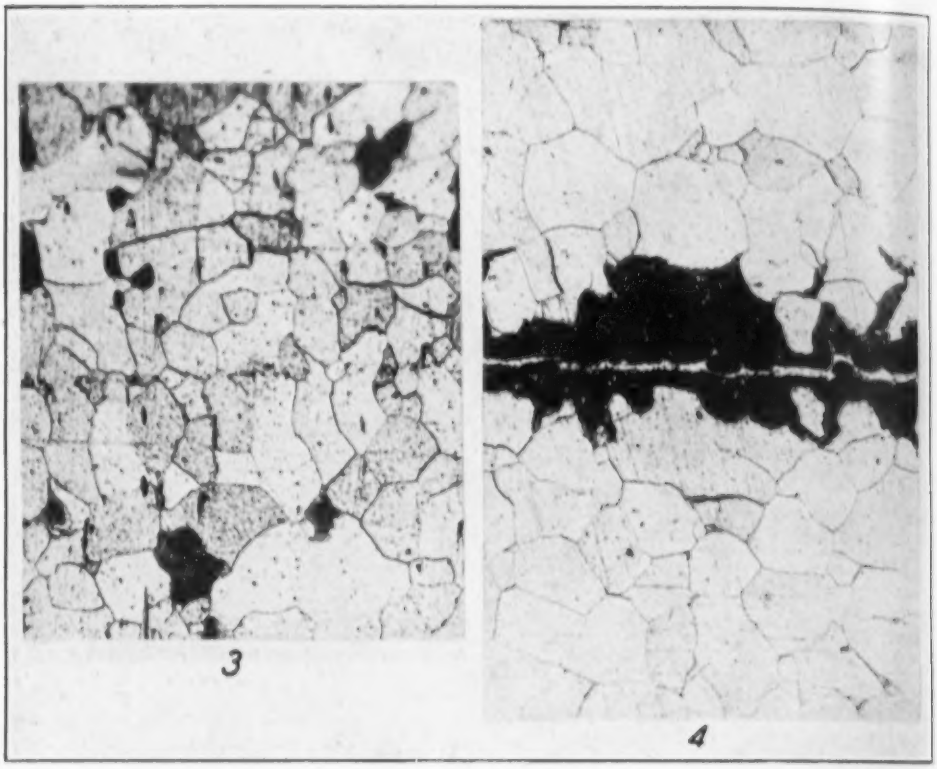


Fig. 3—Shows the Grain Growth Between Two Surfaces of Steel, the Pieces Having Been Put Through a Long Brazing Cycle, Without Copper, Under Pressure of About 100 Pounds per Square Inch.

Fig. 4—Shows a Tightly Assembled Joint, Copper Brazed, Without External Pressure Applied, Showing the Tendency for Grains to Grow Together, and Indicating the Penetration of Copper Along the Grain Boundaries. The Black Grains at the Joint are Pearlite, and not Copper or Copper Alloyed Steel as might at First Glance be Presumed.

into tight crevices quite readily. In fact, the most desirable condition for the copper braze is a steel-to-steel contact at the joint, and a press fit is even better. Test samples always show the greatest strength when subjected to heavy pressures while being copper brazed.

When assembled pieces have clean tight joints there is a distinct tendency for grains of the steel to grow together across the joints during the braze. The effect is very similar to the commonly known grain growth between steel sheets at elevated temperatures, when the sheets are stacked so as to exert pressure at areas of contact. Fig. 3 shows the grain growth between two surfaces of steel, the pieces having been put through a long brazing cycle, without copper, under pressure of about 100 pounds per square inch. Fig. 4 is a tightly assembled joint, copper brazed,

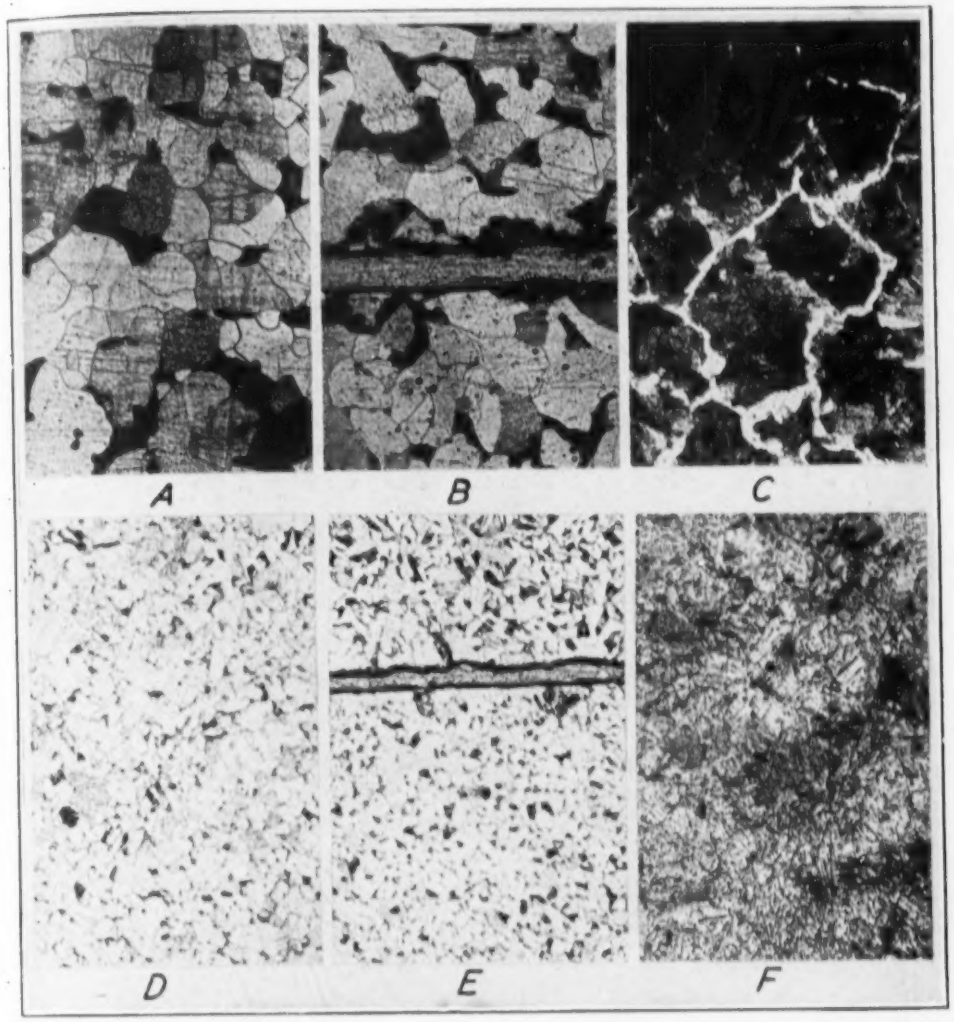


Fig. 5—Shows Grain Refinement of Copper Brazed Specimens. (a) 0.20 C Gear As Brazed. (b) 0.20 C Core of Cam As Brazed. Upper part is cam, Lower part is shaft. (c) High C Case of Cam As Brazed. (d) 0.20 C Gear Copper Brazed Held at 900 Degrees Cent. and Air Cooled. (e) 0.20 C Core of Cam Copper Brazed Held at 900 Degrees Cent. and Air-Cooled. Upper part is cam, Lower part is shaft. (f) High C Case of Cam Copper Brazed and Water Quenched from 785 Degrees Cent. All Specimens × 75.

without external pressure applied, showing the tendency for grains to grow together, and indicating the penetration of copper along the grain boundaries. The black grains at the joint are pearlite, and not copper or copper alloyed steel as might at first glance be presumed.

There are four effects, therefore, which take place in a copper braze, each of which imparts strength to the joint: first, the alloying of copper with the iron; second, alloying of iron with the cop-

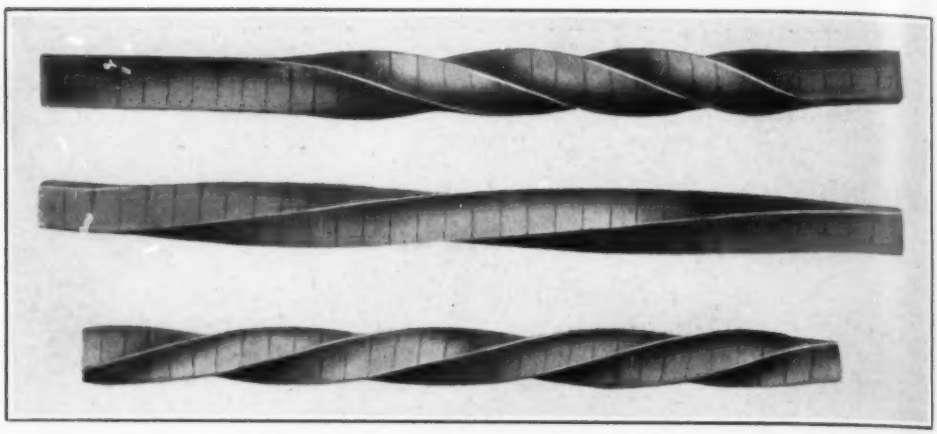


Fig. 6—Showing Test Bars Composed of Series of 0.25 by 0.5 by 0.5-Inch Blocks Copper-Brazed Together. Bars Twisted 180 and 360-Degrees After Brazing.

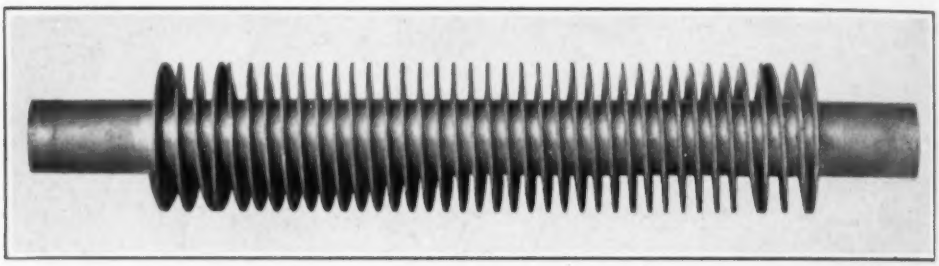


Fig. 7—Steel Tube with Steel Fins, Copper-Brazed in Hydrogen Electric Furnace.

per; third, the penetration of copper along grain boundaries of the iron; and fourth, the growth of iron grains across the joint. The accumulation of these effects produces a union which in many instances, will be as strong as the iron itself.

A rather coarse crystalline structure is usually obtained when passing steel parts through the brazing cycle, because of the relatively high temperatures used (about 1150 degrees Cent.), but in most cases this is of little importance. The parts are dead annealed and ductile as they come from the furnace cooling chamber, and unless they are to be subjected to shocks, there is seldom need for further heat treatment. In event that it becomes desirable to refine the grain, the normal structure can be restored by reheating and properly cooling.

Fig. 5 shows photomicrographs of three parts of a camshaft after brazing, and after subsequent reheating to refine the grain: first, at the left, is a low carbon gear as brazed, below it being a photomicrograph of the same piece after heating at 900 degrees

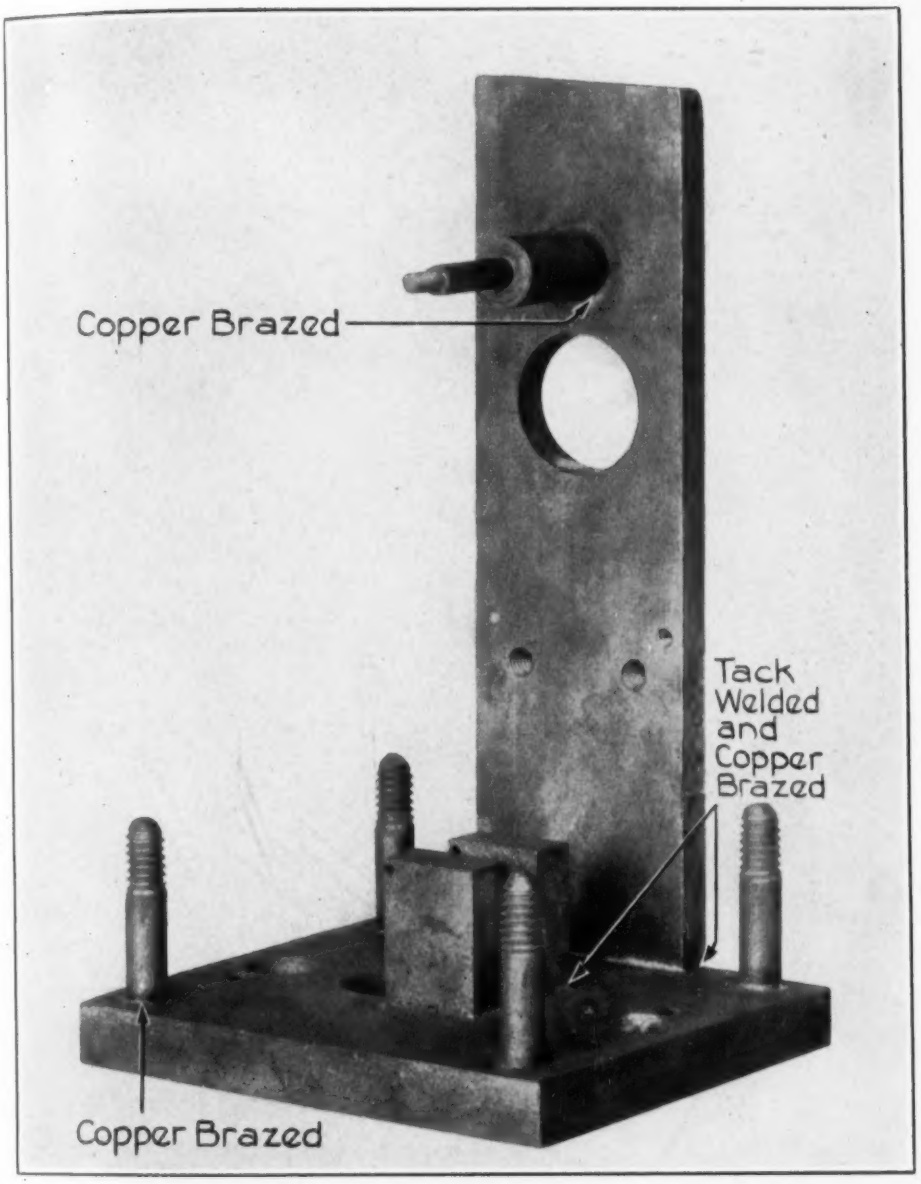


Fig. 8—Thermostat Frame, Copper-Brazed in Hydrogen Electric Furnace.

Cent. (1650 degrees Fahr.) and cooling in air; second is a section of a low carbon cam, copper brazed to a low carbon shaft, beneath which is shown the grain refinement produced by the same heating and cooling used on the gear; third is the high carbon case of a carburized cam as brazed, and below it is shown the effect of reheating to 785 degrees Cent. (1445 degrees Fahr.) and quenching in

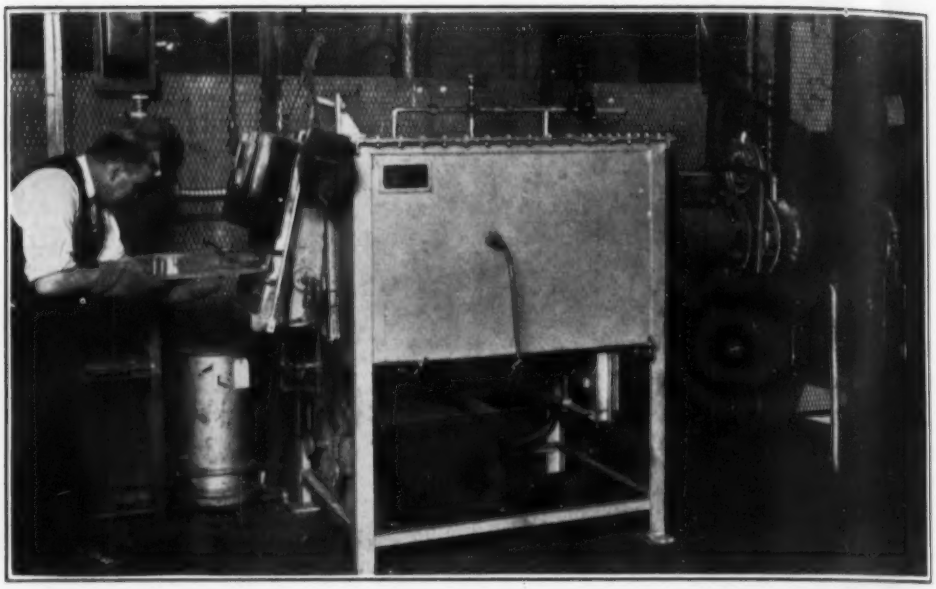


Fig. 9—General Electric 10-Killowatt 220-Volt Hydrogen Electric Furnace, with 6 by 24-Inch Heating Chamber and Molybdenum Wire Resistors, for Copper Brazing Tungsten Carbide Alloy Tools.

water. In all of these samples a good structure has been obtained from the reheat.

Surface decarburization from the hydrogen atmosphere is liable to result, if the parts are heavy and require a long soak in the heat, but a thin electroplate of copper over the high carbon surfaces will give adequate protection from such decarburization in most instances. Little trouble is encountered from surface decarburization of light-weight parts, because they do not require long soaking at the brazing heat. Should such trouble occur, however, the decarburized skin can be ground off, leaving metal at the new surface having the original carbon content. The hydrogen brazing atmosphere (molecular hydrogen) has no embrittling or other bad effect from penetration into steel at the elevated temperature. Embrittlement is caused when articles are pickled in sulphuric or hydrochloric acid, due to penetration of nascent hydrogen into the steel, but the two effects should not be confused.

Many suitable applications have been found for copper brazing, among which are: Refrigerator machine parts such as floats, check valves, evaporators and pistons, automobile camshafts, tungsten-carbide-alloy tools, turbine diaphragm steam nozzles, steel tubing, golf club shafts, steam plates, etc. A few illustrations are given

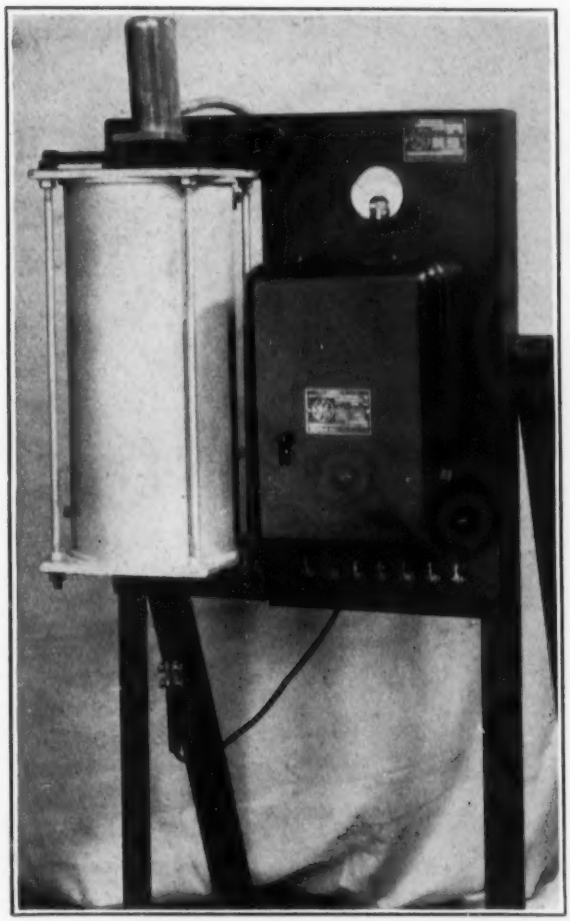


Fig. 10—3.5 Kilowatt 220 Volt Alternating Current Catalytic Ammonia Dissociator, for 50 Cubic Foet Output Per Hour.

herewith to show other examples of copper brazed joints. First is a view of three ½ inch square test bars, Fig. 6, made up of a series of steel blocks ¼ inch thick. After the blocks were copper-brazed together, one of the bars was twisted on its axis 180 degrees and two of them were twisted 360 degrees. The steel in the bars stretched enough to allow twisting, but the brazed joints resisted the tendency to shear.

Fig. 7 shows a steel tube upon which is copper-brazed a spiral steel fin. The assembly is accomplished by tack welding the fin at the ends in the winding process, to keep it in place. Copper wire is then wound spirally between fins and the piece is ready to go into

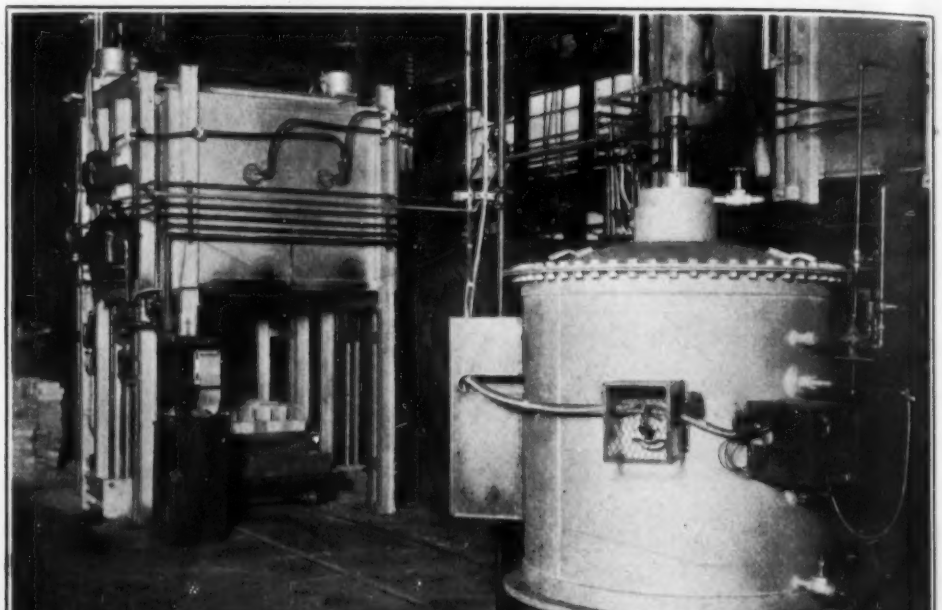


Fig. 11—Electric Annealing Furnace and Electrolene Producer.

the furnace. Such tubing is used in heat transfer apparatus such as condensers.

A thermostat frame is shown in Fig. 8. The four studs are pressed into holes in the base, and a stud is pressed into the back plate. The back plate, in turn, is pressed into a notch in the base and tack welded. A block support is also tack welded to a base. Tack welding of this sort is frequently resorted to, as a means of holding parts together in the furnace. Copper wire and paste are then applied near the joints, and the preparation is complete. An inexpensive, strong, and neat frame is the result.

The design of furnaces operating at the relatively, high copper brazing temperatures, with inflammable gas atmospheres, involves problems not ordinarily encountered in furnace building. Development of suitable furnaces has gone hand in hand with development of the copper brazing process and its application to production needs, and the characteristics of such furnace design are now well known.

All metals under stress, even the nickel-chromium alloys, are at a particular disadvantage within copper brazing furnaces, because

they have little strength at such elevated temperatures. Care is required in designing supports, in order to distribute the load properly, and practical experience with such problems has proved invaluable to designers.

The hydrogen atmosphere within electric copper brazing furnaces protects supports and work from becoming oxidized, and allows the use of nickel-chromium ribbon resistors operating at high watt densities.

Since hydrogen is the lightest substance known, provision must be made to prevent it from escaping in large quantities when the furnace is opened to receive or discharge work. Furnaces with large openings are always charged from the bottom, usually by means of elevators, while small furnaces can sometimes be operated satisfactorily with properly designed sliding or hinged doors at the front and back ends. Pilot flames of illuminating gas are kept burning at all openings, such precaution serving to ignite and burn escaping hydrogen with air as they come into contact with one another, rather than to allow the hydrogen to diffuse and possibly form mixtures which might ignite with violence. Properly constructed brazing furnaces are entirely safe to operate.

Small, compact and inexpensive furnaces have also been designed using molybdenum wire resistors. The older furnaces of this type were made with resistors wound about a tubular alundum muffle, and work was passed through the inside of the tube. Such furnaces are still applicable for some purposes. A later development, the furnace shown in Fig. 9, has molybdenum wire resistors in sinuous loops, wound on removable alundum slabs placed on the side walls. This arrangement provides direct heat to the work, without the intervening muffle. The furnace is rated 10 KW, 220 volts, single phase, and is used for daily production in the copper brazing of tungsten-carbide-alloy tips to nickel steel tool shanks.

In view of the expense involved in the purchase of tanked hydrogen and the high cost of small hydrogen generating plants, new apparatus has been developed for producing suitable gases for copper brazing furnace atmospheres at low operating cost and low initial cost. One process is the dissociation of anhydrous ammonia, and the other consists of reforming hydrocarbon gases, such as city gas. It is not necessary that copper brazing atmosphere be pure hydrogen, although it is essential that they have reducing properties.

Nitrogen, being inert, acts only as a diluent, and can be present in fairly large proportions with no ill effects. Carbon monoxide, having reducing properties, makes a satisfactory addition to a brazing atmosphere. Installations of large copper brazing furnaces usually include a gas generating equipment of one of the above mentioned types, although it is often economical to operate a small furnace, such as that shown in Fig. 9, with tanked hydrogen.

Fig. 10 shows a small ammonia dissociator, rated 50 cubic feet per hour, suitable for furnace Fig. 9. Fig. 11 illustrates an electrolene producer, used in reforming city gas for atmospheres in large electric furnaces, rated 400 cubic feet per hour.

In conclusion it may be said that while copper brazing, from the nature of the process, may not be suitable for some classes of work, there are many instances where it far surpasses other ways of making joints, and it makes feasible certain assemblies that would be wholly impracticable by any other means. Economies have often been shown by the process, and usually one or more of its features such as appearance, strength, gas tightness, ductility, good heat conductivity, etc., makes it particularly desirable for certain applications.

DISCUSSION

Written Discussion: By C. L. Pfeiffer, manufacturing department, Western Electric Co., Hawthorne Station, Chicago.

The process of copper brazing in an atmosphere of hydrogen described by Mr. Webber is no doubt one of quality production. Nearly everyone who has examined the products manufactured by this process of joining materials will agree with such a statement. In addition one is able to design parts which cannot very well be made up by any other method of manufacture. Although the process has some economic limitations, there are many apparatus assemblies which not only can be manufactured more easily, but also more economically.

We would, however, be pleased to hear Mr. Webber discuss the matter of safety in connection with hydrogen furnace operation. As soon as hydrogen is mentioned engineers think of the explosive dangers attending its use. At what time during the cycle of operation is there the greatest danger? What special features in furnace design make for safety? What special precautions are necessary in starting and stopping furnaces? Are the hazards for continuous furnaces less than those for single chamber furnaces? What accidents, if any, have occurred during the last few years and what precautions have been taken to prevent their reoccurrence. What would one do if the supply of both hydrogen and nitrogen were cut off.

We do not believe that the process is particularly dangerous, but we do think it is very important to convince others that the hazards are small and that only ordinary precautions are sufficient to insure safety.

Written Discussion: By Floyd C. Kelley, research laboratory, General Electric Co., Schenectady, N. Y.

Mr. Webber has given a very concise picture of the copper brazing process in hydrogen and its applications. It should stimulate interest in this field and result in broader application.

I can not help thinking that this process is just on the verge of a great development, because in the last few years we have demonstrated in our own factory that furnaces can be designed to handle mass production. The outstanding example is the production of the General Electric refrigerator. During the ten years which the large commercial types of furnaces have been in use, there has not been a single casualty, or serious injury to any employee. We know hydrogen is an explosive gas, but when handled properly is absolutely safe. There are many other things employed in industry which, if not handled properly, are much more hazardous. Greater development may also be anticipated through wider distribution of the apparatus and future changes in design of the hydrogen furnaces.

It might be of interest to examine some data on standard test specimens of S. A. E. 2335 steel which have been copper brazed. The following are the results of tensile tests made upon a series of such samples:

No.	Tensile strength Pounds per square inch
1	69,000
2	71,200
3	74,600
4	69,400
5	74,500
6	80,300
7	72,400
8	78,000
9	78,000
10	71,700
11	86,000
12	63,000
13	75,200
14	77,300
15	73,300

Tensile tests conducted on blanks or samples which have been put through the same heating cycle but not copper brazed are given below:

No.	Tensile strength Pounds per square inch
1	94,000
2	93,000
3	93,500

It is obvious that these joints have about twice the tensile strength of annealed copper. This increase in strength is probably due to several things such as the accuracy of the fit of the joint and the time and temperature of the heating cycle which determines the amount of steel dissolved in copper of the joint. The pressure of one piece of steel against another is also a factor because frequently grain growth takes place across the joints

before the melting point of copper is reached. When such samples are broken, masses of steel are pulled out of the specimen on one side or the other of the copper brazed joint.

Frequently when nickel steel has been brazed to carbon steel, the rupture has taken place entirely outside of the copper brazed joint through the carbon steel, showing that under certain conditions the joint is stronger than the steel itself.

C. B. SADTLER:[1] There is one point which aroused my curiosity in connection with Fig. 4 which appears in the paper. The title reads, "The Black Grains at the Joints are Pearlite and Not Copper." I am just wondering if the pearlite refers to the appearance of a microconstituent resembling pearlite as is ordinarily spoken of in steel metallography, or whether it is actually that well-known pearlite. Is it possible for carbonaceous material to be trapped in joints and exert a carburizing influence producing pearlitic areas?

H. M. WEBBER: There is very little probability of the occurrence of such a carburizing influence, because contact surfaces within joints are ordinarily machined and cleaned before assembly. If a small amount of carbonaceous material should be present, it is likely that it would be taken up by the hydrogen.

Author's Closure

Mr. Kelley has ably expressed my feeling concerning the safety of using hydrogen gas within furnaces for atmosphere. A few additional comments might be helpful.

In the Schenectady factory of our organization, there is used for industrial applications approximately 3,000,000 cubic feet per month hydrogen and 1,000,000 cubic feet per month electrolene. Of this immense volume of gas, about 1,000,000 cubic feet hydrogen and 500,000 cubic feet electrolene is used monthly for furnace atmosphere applications.

There are between 25,000 and 30,000 employees in the above mentioned factory, and you can be assured that the management would not sanction the use of hydrogen atmosphere furnaces if they considered the hazards as great. We have never had a serious accident, and I do not think we ever will have one.

The most dangerous time to operate a hydrogen furnace is while starting it up and shutting it down. Before starting up a large furnace of this type, a good percentage of oxygen is usually present in the chamber, and it is our standard practice to first purge the chamber with nitrogen before admitting hydrogen. Heat can be on in the furnace during this purging period, and when hydrogen is finally turned into the chamber, possibilities of forming an explosive mixture are remote because of the absence of oxygen.

Special features in furnace design making for safety could be followed. All of our older furnaces were designed with explosion proof shells, but experience has taught us that the additional expense necessary to insure

[1]Metallurgist, Barber-Colman Co., Rockford, Ill.

this feature is unwarranted in most instances. Test outlets are always provided at suitable places in the furnace shell to permit taking of gas samples. Elevators and doors usually are constructed so that they will give relief to high internal pressures if ever necessary.

The continuous type furnace having cooling chamber connected with heating chamber, requires slightly more care in operating than the single chamber type, because of the possibility of forming an explosive mixture in the cooling chamber which could then be ignited by hot work coming from the heating chamber. However, by constructing the cooling chamber with gas tight shell, and burning illuminating gas pilots at all openings, presence of dangerous mixtures is avoided. Any oxygen directly entering the hot zone will immediately burn with hydrogen, obviating the formation of explosive mixtures.

For emergency purposes, it is customary to pipe an illuminating gas supply to the furnace chamber, so that in event hydrogen and nitrogen supplies fail, an inflammable atmosphere can be maintained. Recently we had occasion to change piping two different times on a furnace which was in operation, and no emergency line was available The simple procedure of closing all furnace openings and shutting off gas supply was used—the shut-down period being one-half hour in the first instance and one and one-half hours in the second instance. Hydrogen remained within the furnace during these intervals, and there was no indication of danger.

Mr. Sadtler asks whether the black grains at the joint merely have the pearlitic structure, or if they are actually pearlite. It has been observed under the high power microscope that these grains have the lamellar pearlite structure, but whether they actually consist of the well-known pearlite, I should hesitate to say at this time.

RELATIONSHIP BETWEEN WELDED-ON OVERLAYS AND HEAT TREATMENT

By Miles Catlin Smith

Abstract

This paper outlines the procedure and results of a series of investigations covering a period of some eight years and designed to ascertain the value of welded-on overlays as a surface preparation. These experiments, conducted in the field and in the Stoody research laboratories and others, have, above all else, clearly demonstrated the close relationship between welded-on overlays and the heat treatment of metals. By properly combining welded-on overlays and heat treatments, it is possible to sponsor greater economies in machine and machinery construction and fabrication as well as considerably diminish the obsolescence of machine and machinery unit parts, particularly such parts as are subjected to most severe service and wear.

Definition of Welded-On Overlays

STRICTLY speaking, a welded-on overlay is any metallic layer or overlay that has been deposited in place by either of the two autogenous fusion welding processes. However, there seems to be no apparent reason why a metallic surface should be overlaid with a metallic layer quite similar in both physical and chemical properties to itself unless it be to alter a mistake or replace metal worn away in service, so welded-on overlays have come to mean overlays of metallic alloy quite different in character from the metal overlaid.

Approximately eight years ago, Winston F. Stoody asserted that the useful life of oil-well drilling bits could be considerably lengthened by welding on a layer of deposit from an alloy steel welding rod. This was the first welded-on overlay and its reception was none too cordial, nor was its adoption any too prompt. Autogenous fusion welding, as a process, was only mildly successful, at the time, and the linking up of this new procedure with autogenous fusion welding con-

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. The author, a member of the society, is associated with the Stoody Co., Whittier, California, as manager of sales promotion. Manuscript received June 3, 1930.

tributed very little to a cordial reception. Previous to this time, the practice of rebuilding oil-field drilling bits by welding on a soft steel deposit or layer had been successfully tried, although it was not yet in general use, so the alloy overlay was a quite natural development.

This first welded-on overlay material, which soon became known as a "hard facing", was marketed as a composite welding rod, comprised of a soft steel sheath or outer covering in which was held an unfused mixture of manganese, chromium, silicon, iron and a small percentage of zirconium, all mixed with a suitable binder. When this welding rod was utilized as the positive electrode of an electric welding arc or was melted down with the oxyacetylene torch, it formed a deposit which cooled to a hard, abrasive resistant mass and, when this mass was utilized as an overlay for oil field drilling bits, the bits were found to resist wearing away in service many times their unoverlaid capacities. This was the starting period of greater economies in oil field drilling, but, still most important, it was the start of welded-on overlays.

During the succeeding eight years, Stoody Company, of which Winston F. Stoody is president and general manager, and other manufacturing institutions have expended a large sum of money on research and development work with the result that they are now marketing welded-on overlay materials to many individual lines of industry and have several other materials perfected, awaiting a need which is in the offing. Their experience has proven that individual applications are best fulfilled with overlay materials designed for each particular type of application. No one alloy can well have every desired property to meet any and all conditions and requirements.

Since the present-day function of welded-on overlays is to combat abrasion or, in rare instances, combat corrosion as well as abrasion, and since hard substances have been found to resist wear much better than the present-known softer substances, all of the welded-on overlay materials, so far developed, have formed an overlay with a characteristic hardness. The greatest drawback to the development has been that hardness is quite naturally accompanied by a certain amount of brittleness and brittleness is not, at all times, a desirable property in a welded-on overlay. Consequently, the ultimate aim has been to attain the maximum hardness up to the point where the accompanying brittleness would not be damaging.

While this struggle for hardness without brittleness was in process there was little opportunity for a broad adoption of welded-

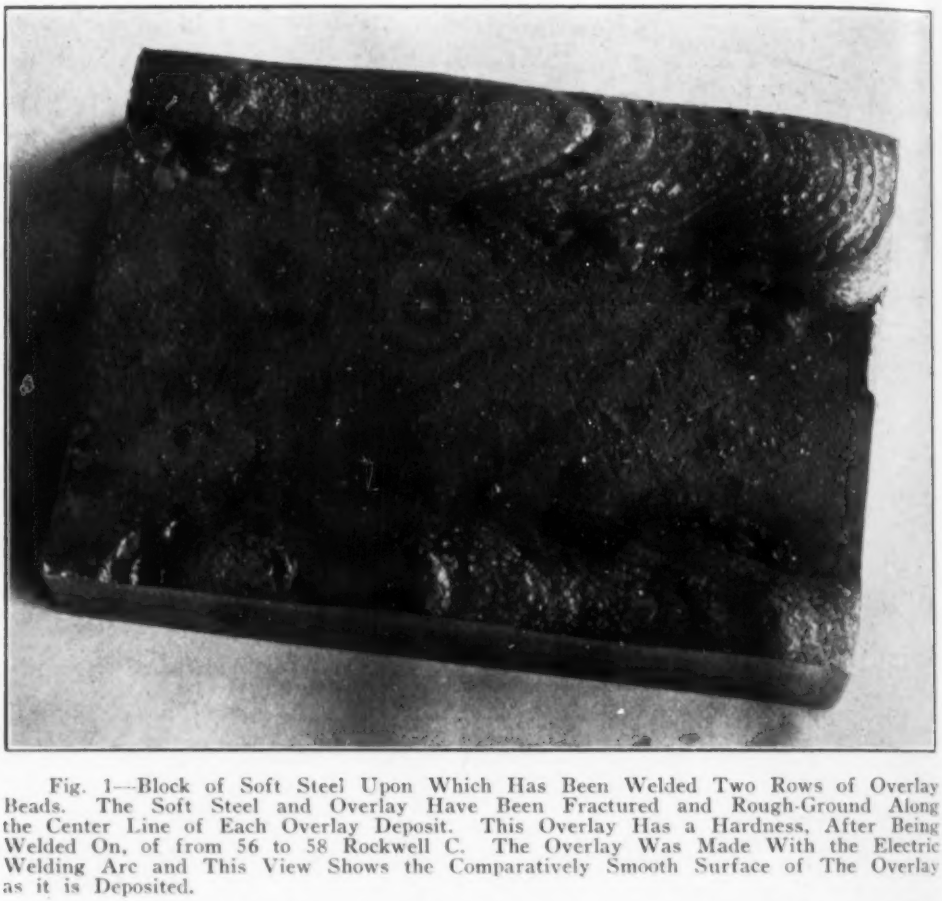


Fig. 1—Block of Soft Steel Upon Which Has Been Welded Two Rows of Overlay Beads. The Soft Steel and Overlay Have Been Fractured and Rough-Ground Along the Center Line of Each Overlay Deposit. This Overlay Has a Hardness, After Being Welded On, of from 56 to 58 Rockwell C. The Overlay Was Made With the Electric Welding Arc and This View Shows the Comparatively Smooth Surface of The Overlay as it is Deposited.

on overlays. Metallic surfaces could be glazed so that they would resist certain kinds of wear, but such overlays fell far short of fulfilling all types of requirements. It was only with subsequent developments which demonstrated the possibility of attaining resistance to abrasion with neither extreme hardness nor damaging brittleness that welded-on overlays became separated from the first-known "hard-facings" and began to show the possibilities of quite universal adoption.

During these years of development, it was found that alloys which were determined some years before the announcement of welded-on overlays could be utilized in the overlay procedure. For the most part, these alloys were the result of a search for metal cutting tools and, previous to their use as overlays, were never deposited by the autogenous fusion welding process. However, experiment proved that they could be welded on and they were soon adopted in the newer procedure.



Fig. 2—View of Rough-Ground Section of Overlay Shown in Fig. 1. The Overlay is Easily Distinguishable as Well as the Uneven Boundary Line Between the Overlay and the Soft Steel is the Line of Amalgamation Between the Two. It is Evident That the Overlay Has Been Completely Fused With the Metal Overlayed.

The welded-on overlay available today is, in reality, a metallic alloy with properties which will best meet and fulfill the definite need—properties which could not be obtained through any sort of conditioning or treatment of the parent metal overlaid. In a word, welded-on overlays make it possible to weld on a surface as and where desired.

The Metallurgy of Welded-On Overlays

The metallurgy of welded-on overlays is most interesting. W. F. Stoody, who pioneered the field, believed that such overlays should attain their resistance to abrasion by proper alloying and proper chemical reactions rather than by the institution of known hard metallic elements or substances. Since it was quite evident that welded-on overlays would be most applicable to use with iron or steel and since a complete amalgamation with the metal overlaid was all-essential, Mr. Stoody built his welded-on overlay materials on an iron base and added the necessary metallic elements to the iron base so that the resulting alloy deposited would, in reality, be an iron or steel alloy.

Other metallurgists, working on the development of welded-on overlay materials, have, from time to time, resorted to the metallic element tungsten and its carbides or have substituted cobalt, nickel or other metallic elements for their base metals and have eliminated all or nearly all iron.

All have been more or less successful, yet all are far from the

acme of perfection expected in later development. The original theory advanced by Mr. Stoody has proven most practical in many applications, particularly so insofar as welded-on overlays for more general adoption are concerned. The necessity of most complete amalgamation between the metal overlaid and the overlay is quite apparent and, while Stoody Company do utilize tungsten carbide in two of their products, tungsten carbide, by its characteristic high melting point and brittleness, is not, at all times, a desirable component of a welded-on overlay. The true overlay is the one which is abrasive resistant, if necessary, corrosive resistant, not exceptionally hard nor exceedingly brittle and one which will actually unite with the metal to which it is applied, so that it will remain in place throughout its useful life. The overlay material must, as well, lend to ease of application with either the electric welding arc or the oxyacetylene torch or both. Furthermore, the metal overlaid must not be damaged or weakened in the process.

Another phase of welded-on overlay utilization which is often overlooked is the fact that either the electric welding arc, or the oxyacetylene flame generate a heat of some 6000 or more degrees Fahr. and, in the ordinary autogenous fusion welding operation, the heated mass is fully exposed to the action of atmospheric agencies. This fact automatically eliminates many metallic elements from consideration as welded-on overlay materials; either they would be too greatly affected by atmospheric agencies or they would volatilize under the influences of so much heat. Of late, most successful experiments have been made along the lines of welding in a hydrogen atmosphere, but the present day cost of such a procedure prohibits it from general use.

Determining the proper elements and the proper proportioning of each element for an alloy to be utilized as a welded-on overlay entails the necessity of determining before hand exactly what the alloy will be after it has been exposed to a temperature of from 3500 to 6000 degrees Fahr. under varying atmospheric conditions and with the operation under the control of one who is not versed in metallurgy, perhaps only partially skilled as a welder. That welded-on overlays have progressed thus far is certainly considerable of a metallurgical achievement. The fact that welded-on overlays may be applied without injury to the structure of the parent metal is an equal achievement, but this has partially been made possible by the general progress

in autogenous fusion welding and only partial credit is due the proponents of welded-on overlays.

To list all of the metallic alloys and elements which have proven to be in some measure adaptable to welded-on overlay compounds would be an almost endless task. Tungsten and tungsten carbides have proven useful in producing hardness, but alloys incorporating tungsten have other characteristics which are not at all times desirable in a welded-on overlay for more or less general adoption. Likewise, silicon contributes to hardness and a recognizable proportion of silicon in a ferrous alloy will produce a hard overlay, yet such an overlay may have a tendency toward surface checking and brittleness that will be quite undesirable. Chromium comes under much the same classification as silicon. And so on through the various better known metallic elements and alloys. From the experience gained in the development so far, it has been conclusively proven that a more or less complex alloy of iron, silicon, carbon, chromium and one or two other metallic elements, in such proportionate quantities that each will contribute its maximum in strength, yet very little, if any, of its weaknesses, will form the best welded-on overlay for the general resistance to abrasive wear.

One particularly interesting sidelight on a particular type of welded-on overlay is the fact that the materials marketed are in the form of a fabricated welding rod. That is, they consist of a soft steel sheath in which is held unsmelted a mixture of alloys and metallic and nonmetallic elements as well as chemical reactory compounds. With such a rod it is possible during the welding procedure to produce chemical reactions, in some respects, similar to the thermit process of forming a weld.

Possibilities of General Adoption

Welded-on overlays make it possible to secure almost any desired surface at almost any desired point or over almost any desired area, providing the area overlaid is of a metal which will respond to the autogenous fusion welding process. At the present time, the need seems to be for an overlay which will protect or which will reinforce cast iron, wrought iron or steel. In most instances, the overlay is utilized with either carbon steel or any one of the more prevalent alloy steels. Consequently, in further discussion, only those overlays which may be utilized with iron or steel will be taken into consideration.

In any and every application, the welded-on overlay is used to acquire a surface, consequently, in any and every application, the overlay should be only a veneer. The most commonly used welded-on overlays are applied to a thickness of from ⅛ to 3/16 inch at their thinnest point, that is, if the overlay is to be ground to smoothness, the finished thickness should be no less than ⅛ inch and no more than 3/16 inch. As it is applied, the overlay should consist of a continuous layer of overlapping welding beads.

Autogenous fusion welding, under its present-day impetus, is too well known to merit discussion and, since welding on overlays, as a precedure, follows very closely the general autogenous fusion welding process, it seems unnecessary to detail any of the methods used other than to mention that welding rods utilized as welded-on overlays have relatively higher melting points and melt down less readily than soft steel welding rods. With the electric welding arc, the overlay rod should be used as a positive electrode and with the oxyacetylene torch, the flame should be free from uncombined oxygen and must be played alternately upon the rod and the metal overlaid.

Simplicity of application, as a matter of fact, apparently, is the key note of welded-on overlays. The manufacturers of present-day overlay materials have seen fit to take the burden upon themselves and manufacture for simplicity of application and use.

Wherein Lies the Relationship Between Welded-on Overlays and Heat Treatment

Heat treatment, or the process of preparing the granular structure of metals so that they will better perform in service, may be divided into two distinct and separate divisions. One division includes those processes which have a tendency to harden the surface and the other includes those processes which are designed to produce structural strength throughout the entire part being treated. Experience has proven that welded-on overlays make it possible to eliminate the first division entirely and, instead of heat treating for surface hardness or a purely surface condition, a surface may be welded on which will meet the desired requirements much better than the heat treated surface could.

Take, for example, a machine part, such as a cam or a lever, which, because of its contact with another machine part must have

a hardened surface to resist the wear of friction between the two. The present practice would be to carburize the part to surface hardness. Welded-on overlays may be employed and, with their employment, the part may be designed for strength and heat treated for strength and the hard surface supplied by welding on the overlay—an overlay which makes possible a surface condition unobtainable by any process of heat treatment. Present-day welded-on overlays cannot be readily machined, but they may be ground to smoothness or to the proper dimensions, so their use is almost unlimited for machine parts.

By the use of welded-on overlays it is both possible and practical to redesign machine parts for greater strength. Furthermore, welded-on overlays could be responsible for less weight and, still more important, welded-on overlays could be responsible for greater economies in machine construction and fabrication, to say nothing, of greater economies in maintenance. Once an overlay does wear through, it is easily replaced without a single change or alteration in the part overlaid.

Insofar as actual tempering or heat treating for a hardness through the piece are concerned, welded-on overlays may be employed after heat treatment, either as a protection to the surface or as a reinforcement which will result in longer useful life of the part. Oil field drilling tools are oftentimes hardened as well as overlaid and the results have been found most satisfactory. In any and every event, the welded-on overlay neither affects nor is it affected by the structural condition of the parts which are overlaid. The overlay produces the desired surface and although it actually amalgamates or alloys with the parent metal to which it is applied, only the actual surface of the parent metal is in any way affected in the process.

Perhaps it would be interesting to briefly cite some of the experiences in heat treating machine parts that have been overlaid, that is, subjecting both the parent metal and the welded-on overlay to the same heat treatment following the overlaying process. For the most part, because of their similar analysis the present welded-on overlays respond to the same heat treatment that has been found best for high speed steel. That is, if the overlaid parts be subjected to temperatures of from 1500 to 1550 degrees Fahr., then quenched in oil and tempered to any desired hardness of the parent

metal, the overlay will be found to have acquired some hardness by the process. Whatever results are obtained though, will be of one character for the overlay and another for the metal overlaid. Even though the two have thoroughly amalgamated at their points of contact, under the influence of heating and quenching, the parent metal and the overlay react in accordance with their individual chemical and physical properties. The following outline of one series of tests which were made under the supervision of a consulting metallurgist will give a very good conception of the performance of overlays under heat treatment. The overlay in this particular instance is marketed under the trade name of Borod which after application leaves a deposit of iron-carbon-tungsten alloy in which is intermingled fine granules of tungsten carbide.

The report:—

"Purpose and Scope of Test. The purpose of this investigation is to determine the effects of heat treatment of the base or parent metal upon the deposited Borod. It is intended to cover the material deposited by the two ordinary methods of welding, namely, by the electric arc and the oxyacetylene process, as well as the extreme quenching and tempering temperatures which should ordinarily be used in heat treating the steels commonly used for drilling bits, in the oil industry."

"Methods of Procedure. In order to test the weldability of the material upon different types of steel, four samples of each were cut from a 0.60 to 0.70 per cent carbon bit steel, an oil hardening chromium steel, and a 0.30 to 0.40 per cent carbon steel, used for several types of bits. The Borod was then applied to duplicate samples made by both processes of welding on each kind of steel. They were tested for hardness on the Rockwell machine. The coupons were quenched according to the kind of steel upon which the Borod was deposited, then tested for hardness and finally tempered at different temperatures, and again tested to determine the effect of this process."

"All samples were broken for a comparison of bond to the parent metal, texture of the fracture, and toughness of the material. A casual observation was made as to the comparative effects of grinding upon the wheel and upon the metal itself, comparing the two methods of application. In this connection, for comparison,

Table I
HARDNESS TESTS—ROCKWELL.—C Scale

Sample No.	Method of Welding	Quenching Medium	Quenching Temp. Deg. F.	As Deposited	As Quench	Quenched as Shown and Tempered at 540° F.	600° F.	1100° F.
H-1	Elec.-Arc.	Oil	1450	C-65	C-65	C-67	....	C-63
H-2	Elec.-Arc.	Oil	1450	C-65	C-65	C-66	....	C-60
S-1	Elec.-Arc.	Water	1520	C-63	C-63	....	C-67	C-61
S-2	Elec.-Arc.	Water	1520	C-63	C-65	....	C-66	C-63
C-1	Elec.-Arc.	Oil	1500	C-63	C-63	C-66	....	C-63
C-2	Elec.-Arc.	Oil	1500	C-63	C-63	C-65½	....	C-62
H-3	Acetylene	Oil	1450	C-51	C-51	C-52	....	C-50
H-4	Acetylene	Oil	1450	C-54	C-49	C-47	....	C-50½
S-3	Acetylene	Water	1520	C-50	C-54	....	C-51	C-50
S-4	Acetylene	Water	1520	C-46	C-56	....	C-49	C-46
C-3	Acetylene	Oil	1500	C-51	C-50	C-48	....	C-50
C-4	Acetylene	Oil	1500	C-45	C-52	C-50	....	C-49

In addition to the above tests which were made on welds, made by outside welders, an average of ten readings on samples welded at the Stoody plant, show as follows:

Table II

Method of Application	As Deposited	Quenched at 1500° F. in Oil	Quenched in Oil at 1500° F. and Tempered at 540° F.	1100° F.
Electric Arc	C-61	C-64	C-62	C-56
Acetylene	C-51	C-55	C-49	C-49

we note that Rockwell hardness of tungsten carbide materials such as carboloy is reported as C-74.

It will be noted that the electrically deposited material is considerably harder than that put on by the oxyacetylene process. This fact is undoubtedly due to the greter heat of the arc, which fuses a considerable portion of the tungsten carbide filler, while the acetylene flame merely fuses the parent metal and the tube of the rod, possibly dissolving a small amount of the filler. A comparatively high heat used by the acetylene welder at the Stoody plant did not make a very wide difference in hardness over a somewhat lower temperature used by the welder who had never put on any Borod."

"*Effects of Heat Treatment.* Quenching of the electrically deposited metal had very little effect on the hardness of the material. However, a low tempering treatment, 540 to 600 degrees Fahr. shows a marked increase in the hardness, which is brought down again by a high temper (1100 degrees Fahr.). The ordinary tempering temperature for drilling bits is in the neighborhood of 450 to 600 degrees Fahr."

The acetylene-welded material shows a greater variation in hard-

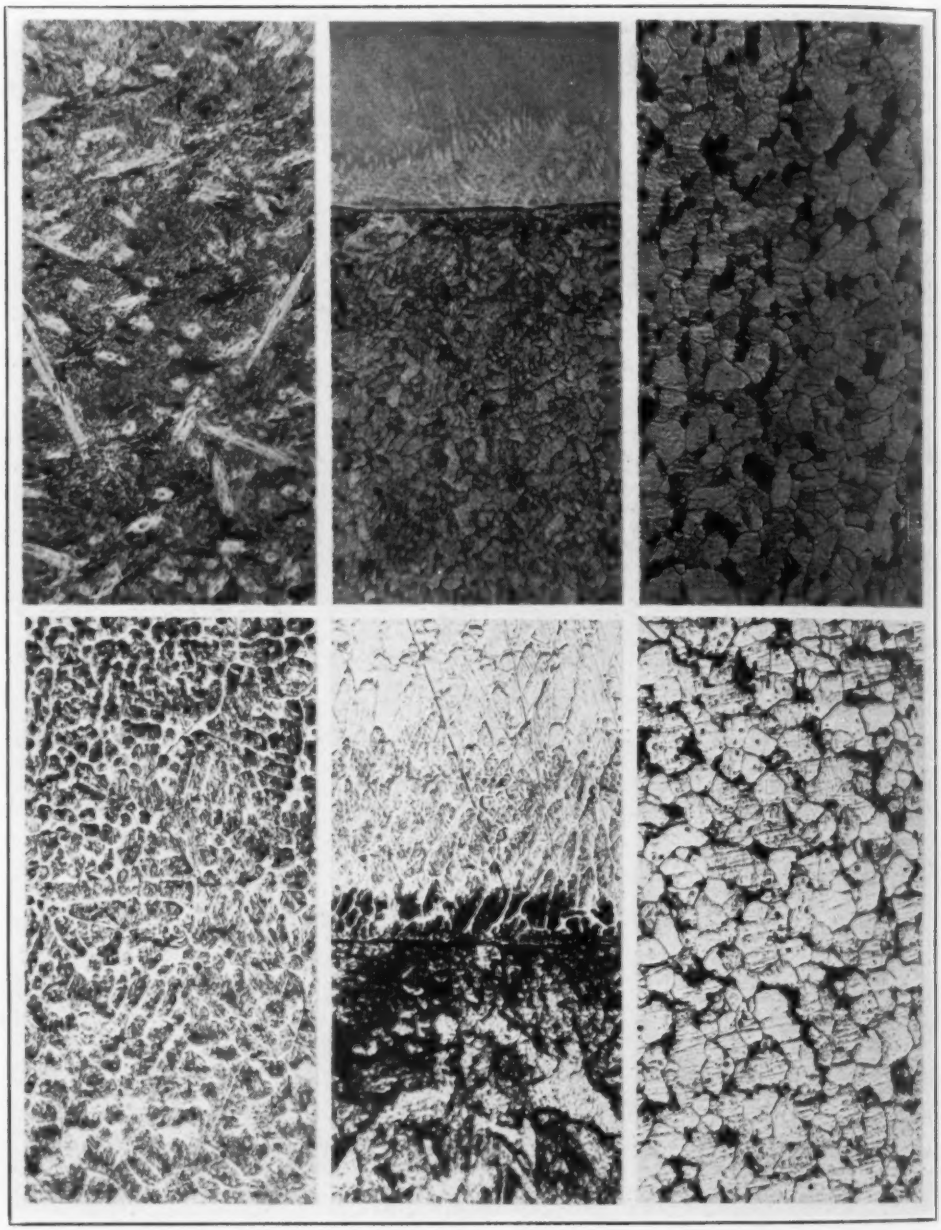


Fig. 3—Photomicrographs of Overlay in Fig. 2. (Top Row) The Left Picture Shows the Structure of the Overlay With its Characteristic Crystalline Structure. This Overlay Deposit is an Alloy of Iron, Chromium, Manganese, Silicon, Carbon and Zirconium.

The Center Picture is Taken at the Line of Amalgamation Between the Overlay and the Soft Steel. Aside from a Tendency Toward Refinement of the Soft Steel Grain Structure in the Vicinity of the Line of Amalgamation, the Soft Steel Structure Remains Unchanged and, Even if it Had Been Heat Treated, the Soft Steel Would Have Remained Structurally Unchanged.

The Right Picture is of the Soft Steel Structure Farther Down in the Piece and Serves as a Comparison With the Soft Steel Structure Shown in the Center Picture. × 100.

Fig. 4—(Bottom Row) Photomicrographs of Overlay Made With Stoody Rod Which Shows a Hardness on the Rockwell C Scale of From 53 to 55. The Left Picture is of the Overlay, the Center is of the Line of Amalgamation and the Right is of the Parent Metal Structure × 100.

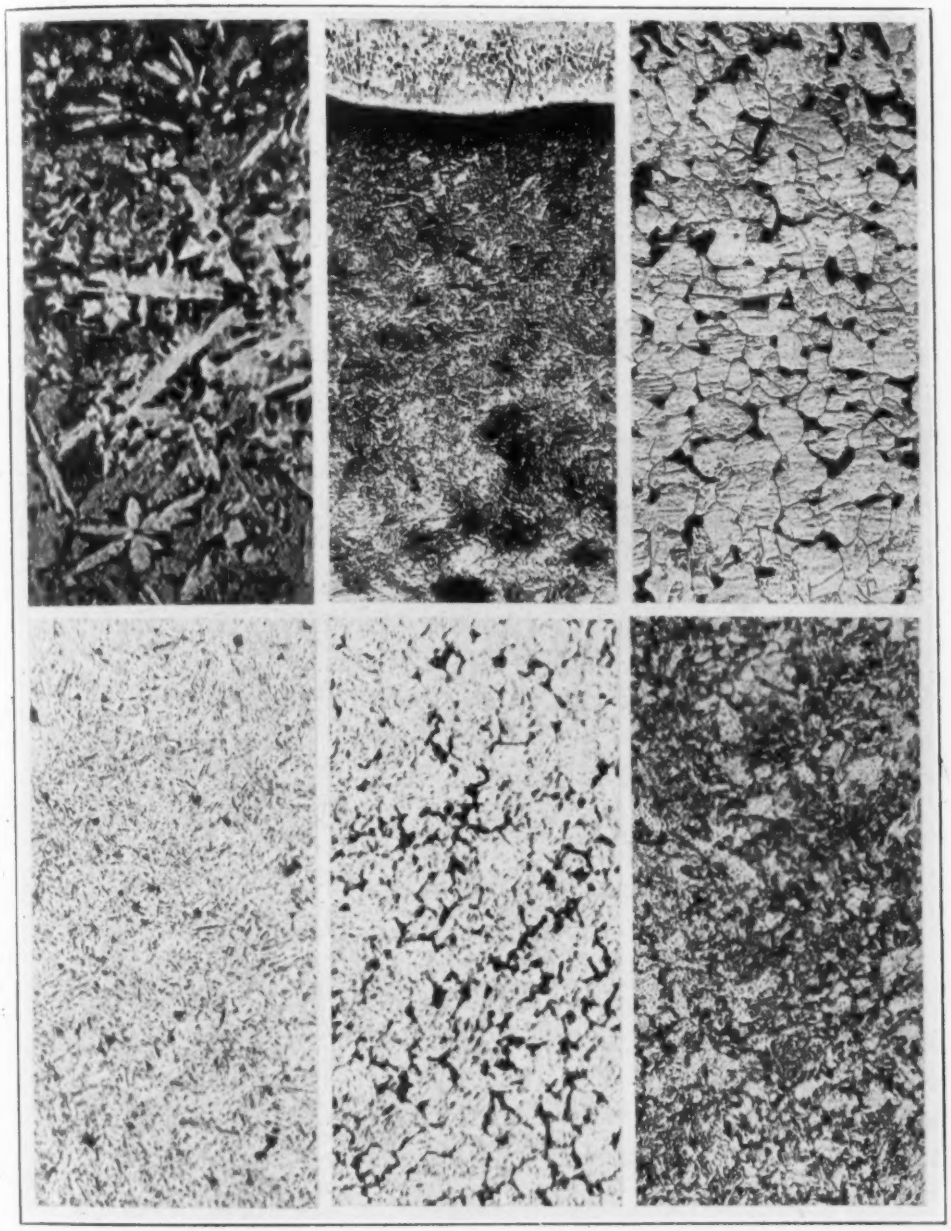


Fig. 5—(Top Row) Photomicrographs of an Overlay Made From Borod. This Deposit Consists of Fine Granules of Tungsten Carbide, Held in Place and Intermingled with a Body of Tungsten-Carbon-Iron Alloy. The Left Picture at ×500 Clearly Shows the Tungsten Carbide Crystals. The Center and Right Pictures are at ×100 and Bring Out the Same Facts Demonstrated in Figs. 3 and 4. The Purpose of Such an Overlay is to Secure Tungsten Carbide Resistance to Abrasion and Eliminate Much of the Tungsten Carbide Characteristic Brittleness.

Fig. 6—(Bottom Row) Photomicrographs of Case Hardened Soft Steel, ×100. The Left Picture is of the Case, the Center of the Parting Line Between the Case and Core and the Right Picture is of the Core. Case Hardening Produces no Definite Line of Amalgamation as does the Process of Welded-on Overlays, but, on the Other Hand, Case Hardening Effects the Structure of the Core as Well as the Structure of the Outer Shell or Case. With Case Hardening, it is Not Possible to Obtain a Surface of One Character and Consistency With no Change to the Metal Surfaced.

ness, both before and after heat treating. This result is quite as would be expected, inasmuch as a great deal less of the tungsten is actually placed in the solution by the acetylene torch. The metal consists of particles of tungsten carbide in a steel matrix which derives its composition from the tube container, the fusible or soluble portions of the alloy filler, and the parent metal which becomes mixed in. Naturally, there is more or less variation in composition and hardness. Some cracking and a tendency to shell off was found in the electrically welded samples, especially those welded at the Stoody Company's shop. No cracking or shelling off was noted in the gas welded samples. The bond on the chromium alloy steel was fully as good as on the carbon steel."

"*Grinding Test.* The behavior of the metal was observed in a general way while grinding down the samples for testing. While both materials will dress an emery wheel, somewhat after the manner of the tungsten carbide inserts, the cutting into the wheel is less pronounced, and the deposited metal can be ground more rapidly, indicating a somewhat less hardness."

"The metal deposited by the acetylene torch appears to resist grinding more than the electrically deposited alloy."

Conclusions

1. Heat treatment does not materially change the hardness of deposited Borod.

2. Quenching may produce cracks in electrically deposited Borod. It appears that arc welding might be preferred where no heat treating is to be done, while acetylene welding is preferable where it is intended to heat treat. It is possible that a change of arc welding practice might adapt the arc welded material to heat treating to better advantage.

3. The comparatively soft matrix deposited by gas welding in which are embedded the particles of tungsten carbide, appears to resist abrasion better than the harder arc welded metal. Further abrasion tests on this point are recommended.

This particular test is cited because the overlay material is an unusual one and affords a particularly good opportunity for demonstrating the wide range of results obtainable through a variation of the methods of application. As marketed, Borod consists of a soft steel sheath or outer covering, with a core of fine granules

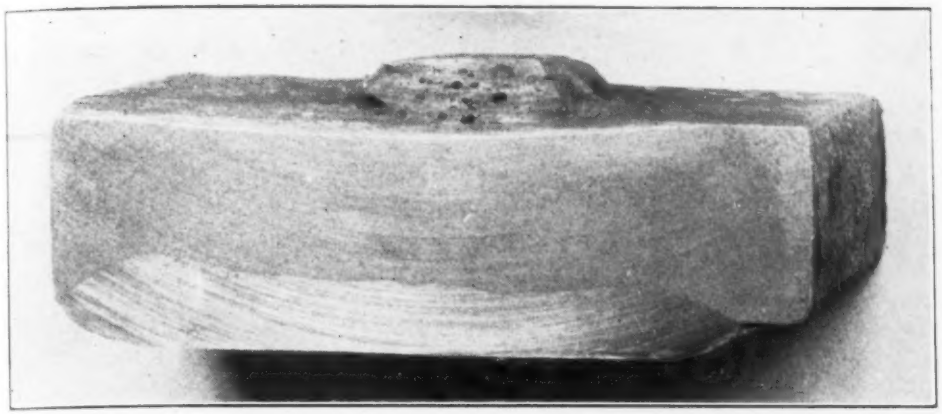


Fig. 7—On One Side of This Specimen an Overlay of Marketable Welding Alloy was Deposited and on the Other Side an Attempt was Made to Overlay With an Alloy Not Properly Compounded for the Purpose. The Piece was Then Fractured Through the Center. The Difference in the Two Deposits is Evident. The Overlay of Marketable Alloy Shows a Grain Quite Similar in Appearance to a Fine Grade of Tool Steel While the Other Deposit is Full of Holes and Far From Satisfactory.

of tungsten-carbide and a relatively small proportion of other ingredients which act as a binder and have little or nothing to do with the character of the deposit when the rod is welded on. Such a material when melted down and allowed to cool forms a deposit fully in accord with its method of application. Other overlay materials are similarly affected but the range of results possible are not nearly so pronounced.

From the cited report it may be seen that the overlay responds to some extent to heat treatment, but has absolutely no effect on the response of the parent metal to which it is applied. Any and all of the deposits mentioned in this report are abrasive resistant and, to all general purposes, satisfactory.

To cite other experiments would be very much of a repetition of the report mentioned and while the variations would not be equally pronounced, variations would exist and could be analyzed just as they have been in the report on Borod.

Observations of Various Applications

All of the foregoing conclusions and recommendations have resulted from microscopic studies of thousands of varied applications of welded-on overlays. By a close examination of the structures and by comparing the photomicrographs of these structures with the photomicrographs of other structures, it has been possible

to prove beyond any question of doubt that the overlays do amalgamate with the parent metal, that they do have a characteristic structure, quite unlike the parent metal and which is demonstrative of their hardness and resistance to abrasion, and, most important of all, the structure of the parent metal overlaid is, to no perceptible extent, altered, even adjacent to the line of amalgamation.

For the most part, the overlays used in the petroleum exploration drilling industry are designed for a purpose not found in the average application of welded-on overlays and both the processes and the overlay materials are not suitable to most general use. In the case of oilfield drilling tools, the service as well as the shape of the tools themselves lend to a type of overlay which would be too hard, too brittle and too difficult to apply for general use.

To be profitably used in general applications, the overlay material must be, to a degree, ductile. It must be easy to apply. Experiences, so far, with tungsten and tungsten carbides, either in their nearly free state or as components of an alloy, aside from in such form as in the Borod mentioned, have been far from universally satisfactory as a general purpose overlay. On the contrary, those overlays which attribute their wear-resisting properties to an alloying of metallic elements with lower melting points have proven quite satisfactory. The latter are much easier to apply, less brittle after they are in place.

Rarely, if ever, the first theories advanced and the first conclusions drawn concerning any new process are found to endure, but, in the instance of welded-on overlays, Winston F. Stoody, the originator of both the process and the first material to be used for the process, advanced, at the outset, theories and principals which stand unshaken today. The more universal the application of welded-on overlays becomes, the more apparent it is that the original theories were the proper ones and the original method of compounding was and is the ideal method.

DISCUSSION

Written Discussion: By W. A. Wissler, Union Carbide and Carbon Research Laboratories, Inc., Long Island City, N. Y.

The early history of any industrial development is always of interest, although it has but little bearing on the present economic value of the process or the materials used. In an effort to increase the accuracy and completeness of the data given, the following should be added to Mr. Smith's paper.

The Electro Metallurgical Co. in 1918 used hard facings of various chromium alloys on machine parts in their own plant, applying them by the oxyacetylene process. Experimental and development work done in 1919 and 1920 showed that Haynes Stellite was suitable for this purpose, and in 1921 the alloy was being tested for several applications. In 1922 Haynes Stellite was being used successfully, one particular instance being the Stelliting of forming dies used in Canton, Ohio, for making railroad creepers, the resulting life being 100,000 pieces from a Stellited die as against 6,000 to 10,000 from the high speed steel dies previously used. From this time on the use of Haynes Stellite as a hard surfacing material spread rapidly. In the fall of 1924 it was introduced as a proven material for the hard facing of fishtail and other bits. One of the early authentic papers on the use of Stellited fishtail bits was written by Mr. Suverkrop, an inspector of the California State Mining Bureau, and published by that Bureau in their March, 1925, Summary of Operations. This paper describes in considerable detail the method of application in use at that time and the results obtained by the use of Stellited and steel bits. Since its first introduction improvements have been made in Stellite to facilitate welding, but the basic composition and the essential properties have remained the same.

As a criticism of a minor point in the paper I would suggest that hardness when measured cold is not a safe basis for the prediction of resistance to abrasive wear. It is well known that most steels can be made considerably harder than Stellite or other hard facing metals, but they do not retain their hardness at high temperatures. There is a large amount of data and several theoretical reasons indicating that the extreme surfaces of materials subject to abrasive wear become highly heated, and obviously the effective hardness of a particular alloy is the hardness measured while under operating conditions. Consequently hardness values of metals designed for resisting abrasion or for cutting tools should be measured within the red heat range, under which condition we find that the cobalt-chromium-tungsten alloys have a higher degree of hardness than any other hard facing metal.

There is reason to doubt the statement that if a ferrous metal part is used the hard facing metal must also be a ferrous alloy. None of the higher grade hard facing metals are as strong as steel and it is unnecessary to have a bond between the two that is stronger than the hard facing metal itself. A bond of ample strength may readily be obtained by any process similar to Stelliting, which, if the oxyacetylene torch is used, produces a straight line junction between the two metals and results in a minimum of interalloying. Regardless of whether or not the hard facing metal has an iron or a cobalt base, interalloying with the steel base will reduce its content of those elements that have been added to increase wear resistance, and hence reduce its effective life and value. The aim therefore would seem to be to endeavor to keep interalloying at a minimum but to insure that the junction between the two metals is equal in strength to the hard facing metal itself.

In regard to the statement that the oxyacetylene torch generates a temperature that would prevent the use of many metallic elements, it should be pointed out that the metals themselves do not reach the flame temperature. Magnesium,

aluminum, and zinc can be welded by the oxyacetylene process. By proper manipulation the metals need be heated only slightly above their melting points.

Author's Closure

Replying to Mr. Wissler's discussion, the early history mentioned by Mr. Wissler is undoubtedly true and it is this author's knowledge that considerable experimenting was done with the material known as Haynes Stellite during 1919-20-21-22 and 23. But it has been an accepted fact that Mr. Winston F. Stoody was personally responsible for the start of what is known as hard-facing in the oil fields and it was only after Mr. Stoody had successfully proven his theories that Haynes Stellite was used with any definite amount of success. Prior to that time the management of the Haynes Stellite Company had apparently thought little of this use and considerable correspondence is now in existence discouraging the use of Haynes Stellite as a welding rod and endeavoring to keep Stellite in its original field—that of metal cutting tools in machine shop practice.

Relative to Haynes Stellite and a process known as Stelliting, this author has never considered Stelliting as belonging to the general classification of welded-on overlays. The present accepted methods of applying Haynes Stellite are discouraged as Stelliting is not a welding process, but quite similar to a soldering or brazing process. In other words there is apparently no complete amalgamation of the Stellite and the parent metal to which it is applied. Rather, there is a congealing between the two metals which is clearly proven by the complete clean line of cleavage easily seen under the miscroscope. However, it is a fact that this Stelliting does produce a surface condition which is quite equivalent to that obtainable by the welding-on of an overlay and as long as the deposit of Haynes Stellite remains in place it will function as a welded-on overlay.

It was the purpose of this paper not to get into any discussion as to the relative merits of any materials or processes, but rather to point out the possibilities of acquiring any desired surface condition by welding on a metal or alloy which would produce that condition. This applies equally well to the much taunted red hardness theory, as well as to any other criticisms brought out in Mr. Wissler's discussion. Furthermore, it does not seem to be essentially important whether they be interalloyed as Mr. Wissler has mentioned or not, as the desired surface can be acquired with, or without interalloying, that is all that is necessary.

Summing up—this author feels that Mr. Wissler's discussion is more in defense of a particular product than as a discussion of the subject of this author's paper.

AN INSTRUMENT FOR HIGH-VOLTAGE X-RAY SPECTROGRAPHY AND RADIOGRAPHY

By Frederick Sillers, Jr.

Abstract

The increasing application of the X-Ray diffraction method to the study of materials of construction has led to the desire for a rapid method of obtaining results. Such a method may involve the adoption of higher voltages than are now generally employed. The present paper describes an instrument for use with voltages in the neighborhood of 200 kv. With this instrument four X-ray spectrograms, of comparable intensities, may be obtained from coarse-grained material such as silicon electrical steel in less than one hour. By rotating the X-ray tube 90° on its axis radiographs may be obtained in the customary manner.

Points discussed deal with strain in the steel, the use of intensifying screens, selection of optimum voltage, and intensity variation.

THE increasing use of the X-ray spectrographic or diffraction method in the study of materials of construction has led to the desire, in some fields, for a more rapid method of obtaining results. Such a method would involve the adoption of higher voltages than are now in general use. Since it has not come to the writer's attention that a specific instrument for obtaining these results has been reported in the literature, in any detail, he believes it might be of interest to describe the present one.

This instrument was constructed for the purpose of studying sheet steel, particularly high silicon steel for electrical uses. It was intended to be used with a standard high voltage X-ray installation of the type ordinarily used in radiography.

A photograph of the instrument[1] (taken at the factory just after its completion) is shown in Fig. 1. Since that time several slight modifications have been made which are described in the text. It will be noted that the device consists essentially of a drum sup-

[1]The instrument was constructed by the Schütte Electric Company of Pittsburgh, according to the plan of the writer. The latter is indebted to Mr. Hermann Schütte of that company for working out the details of construction of the finer parts.

The author, Frederick Sillers, Jr., a member of the society, is research engineer, Follansbee Brothers Co., Follansbee, W. Va. Manuscript received March 4, 1930.

ported horizontally, around the periphery of which are mounted four "diffractometers". The latter serve to define the X-ray beam, support the specimens under examination, and house the photographic films. The X-ray tube, at present of the Coolidge 200-kilovolt deep-therapy type, rests horizontally on sutiable supports inside the drum. The drum is lined with lead ⅛ inch in thickness as is the chamber in which the instrument is housed. A protection afforded by ¼ inch of lead is thus given the operator who remains outside the chamber during operation of the machine. This thickness of lead is considered ample for protection against rays of the hardness produced by 200 kilovolts, the maximum operating voltage. Other precautions were taken in building the instrument such as covering all steel bolts with lead and lapping all seams.

The vertical cylinders visible in the photograph are composition insulators installed for the purpose of shielding the high tension lead-in wires running to the X-ray tube. These are used wherever such high tension wires pass through partitions or come into contact with a "ground".

In the bottom of the drum is an opening, covered by a hinged door, which permits the taking of ordinary radiographs in which case the specimen and film-holders are laid on an adjustable rack beneath. The film-holders, or cassettes, permit filtration of the rays through a lead screen and thus increase the sharpness of the resultant radiograph.

Fig. 2 is a drawing of a cross section of the instrument. The X-ray tube, as previously stated, lies on the longitudinal axis of the drum and the four diffractometers are equally spaced about it within an arc of 100 degrees. The magnitude of the arc was so chosen after inspection of a series of intensity curves determined by Coolidge.[2] In this work the intensity of the X-rays in various planes had been measured and plotted. From his results Coolidge stated: "In the equatorial plane (about the girdle of the tube), the intensity is essentially constant through an angle of about 100 degrees". This is an important consideration in diffraction work if comparable results are to be obtained among a number of specimens examined simultaneously. Other planar, or angular, relationships between the tube and the diffractometers would not have yielded such a favorable arc in which to work.

[2] "High Voltage X-Ray Work," W. D. Coolidge and W. K. Kearsley, Jr., *American Jounnal of Roentgenology*, Vol. 9, No. 2, Feb. 1922, pp. 77-101.



Fig. 1—P
Radiography.

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appliances.

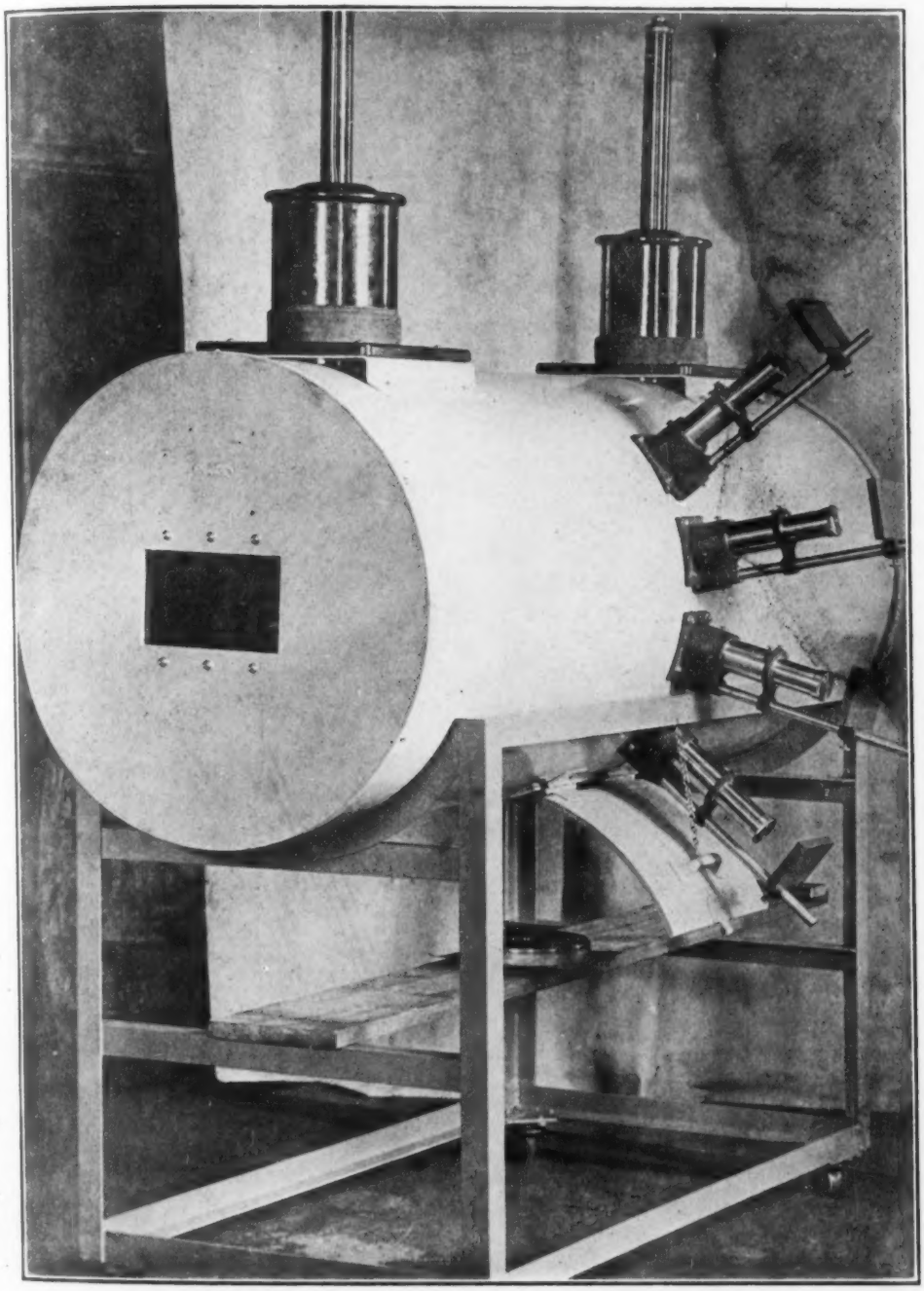


Fig. 1—Photograph of Instrument for High-Voltage X-Ray Spectrography and Radiography.

The clamps for supporting the tube at either end are standard appliances. In the present instances they are mounted on a suit-

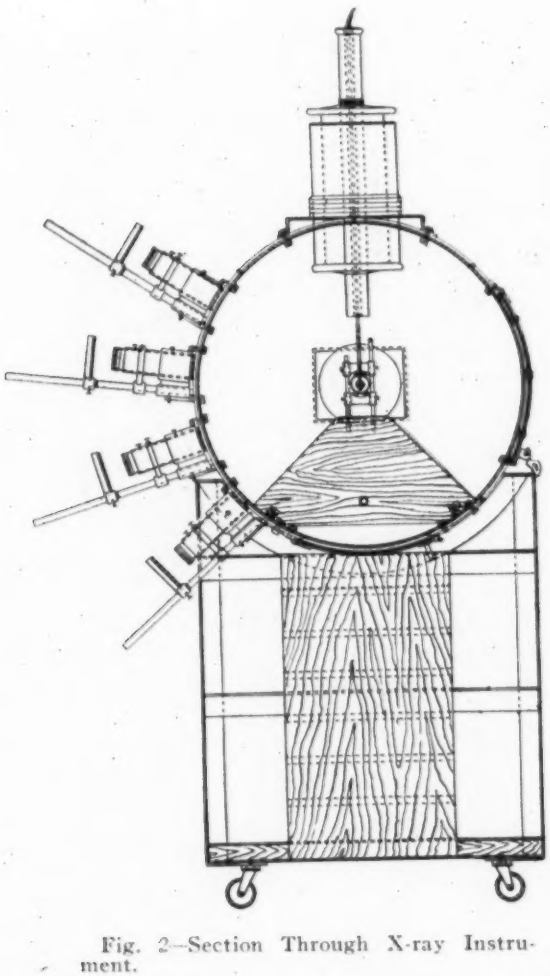


Fig. 2—Section Through X-ray Instrument.

able wooden base coated with clear lacquer.

The supporting standard for the drum is built of welded angle iron and mounted on rollers. The instrument may be moved about with relative ease.

The adjustable rack, added later, for radiographic specimens is of wood. This construction was deemed to be of sufficient strength for supporting such specimens as would be likely to come to the writer's attention.

An idea of the details of the diffractometers may be had from inspection of Figs. 3 and 4. The former is a near view of the four devices mounted on the drum and the latter a view of one of the devices disassembled. The diffractometers consist essentially of two sleeve-fitting, lead-lined brass tubes, in the end of each of which, is

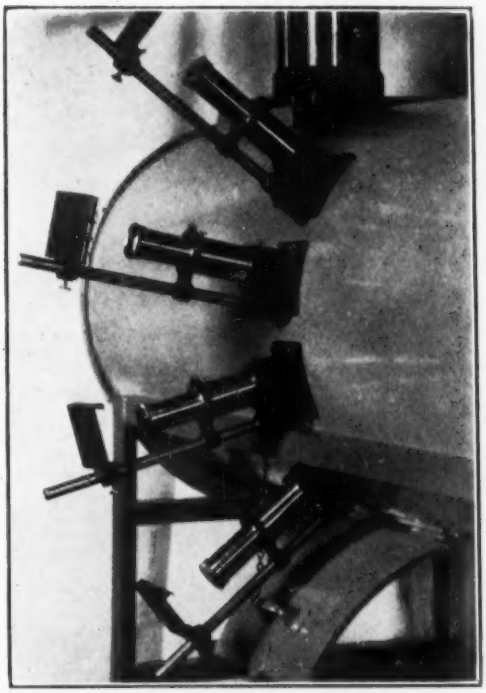


Fig. 3—Near View of Diffractometers.

a lead diaphragm containing a "pin-hole". The diaphragms are mounted in screw caps so as to be readily interchangeable in case of experimentation with different type orifices. The forward pin-hole is 0.0275 inch in diameter, and the rear orifice ⅜ inch in diameter. Experience showed that a large hole was desirable for admitting the X-rays to the diffractometer. In practice, the writer finds it best to employ the "telescope" of the diffractometer completely collapsed in order to take advantage of the greater intensity of radiation obtained by operating closer to the target of the X-ray tube. The parallelism of the rays is sufficient under these conditions for ordinary work.

The film-holders are standard 3¼ by 4¼ inch devices. They are supported in guides and held in place by leaf springs.

Reference to Fig. 2 will indicate that the X-ray tube is capable of rotation about its longitudinal axis when the clamps supporting it at either end are loosened. Use is made of this feature in changing from spectrographic to radiographic work. It will be recalled that the target, or anode, of a Coolidge tube makes an angle of 45 degrees with the longitudinal axis; hence, for spectro-

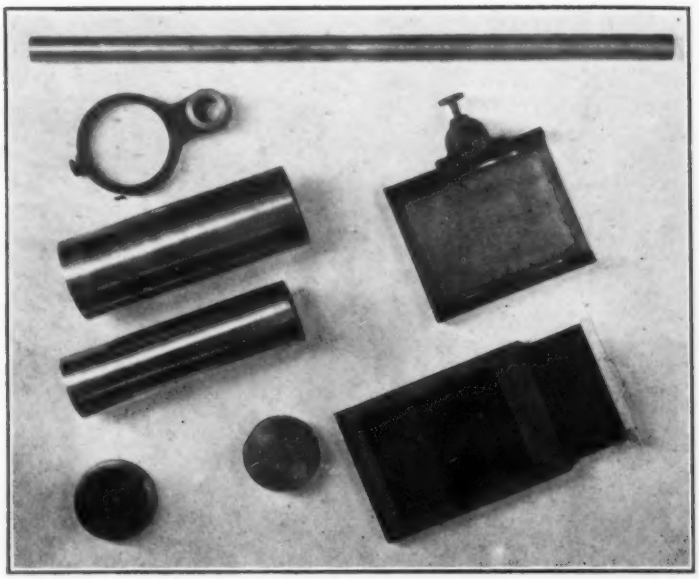


Fig. 4—One of the Diffractometers Disassembled.

graphic work, the target is made to face a point at the center of the arc subtended by the diffractometers, and for radiographic work it is pointed directly downward, illuminating any object placed on the rack beneath. One end of the drum contains a window of lead glass used for sighting, with the aid of a mirror, on the tube in operation.

It was found during the course of some early experiments that films from the two upper diffractometers were fogged. This fogging was traced to leakage of "reflected" X-rays from the high tension insulators. By introducing sheets of lead into the path of the radiation it was found possible to produce definite "shadows" on the photographic films and to identify the fogging as leakage from the insulators. This difficulty was corrected by supplying lead bonnets to the two upper diffractometers. These completely covered them and were removable.

Fig. 5 is a view of the X-ray chamber with the instrument in position for operation. The control stand at the left of the door of the chamber and the adjustment regulators on the wall are used in routine operation. During voltage calibration, tube-current adjustment and the like, it is necessary for the operator to remain inside the chamber for short periods of time but the X-ray dosages received, it is believed, are within the limits of safety due to the

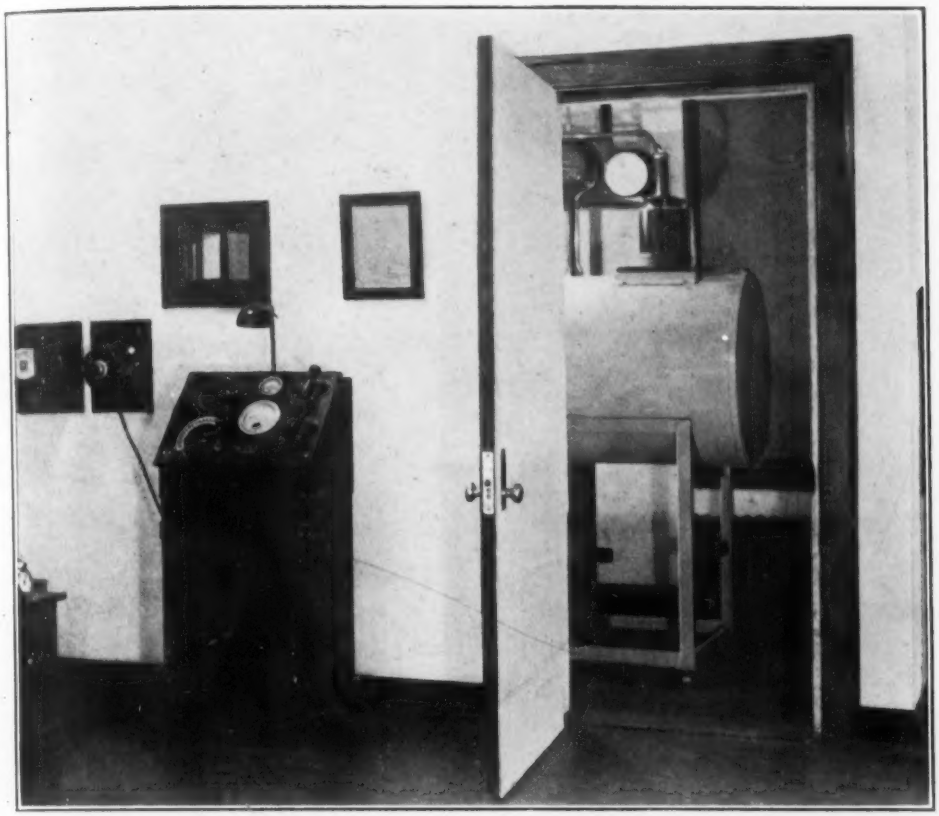


Fig. 5—Photograph of X-ray Chamber with Instrument in Position Ready for Operation.

protection afforded by the instrument itself. The meters, partially visible through the open door, are observed during runs through the lead glass window above and to the left of the control stand. The voltage transformer and rectifier are in the basement directly beneath the X-ray chamber.

Diffraction Patterns

Typical diffraction patterns of silicon steel obtained with the present apparatus are given in Figs. 6 to 8. Fig. 6 is illustrative of a characteristic application of the X-ray diffraction method in the study of this material. The presence of strain, introduced by hot rolling, in some experimental specimens of high silicon electrical sheet is clearly indicated by the presence of asterism in the diffraction patterns. The condition of the initial material (6a) is that of well annealed, coarsely crystalline steel. Hot rolling to various degrees of elongation (Fig. 6b and 6d) has introduced definite

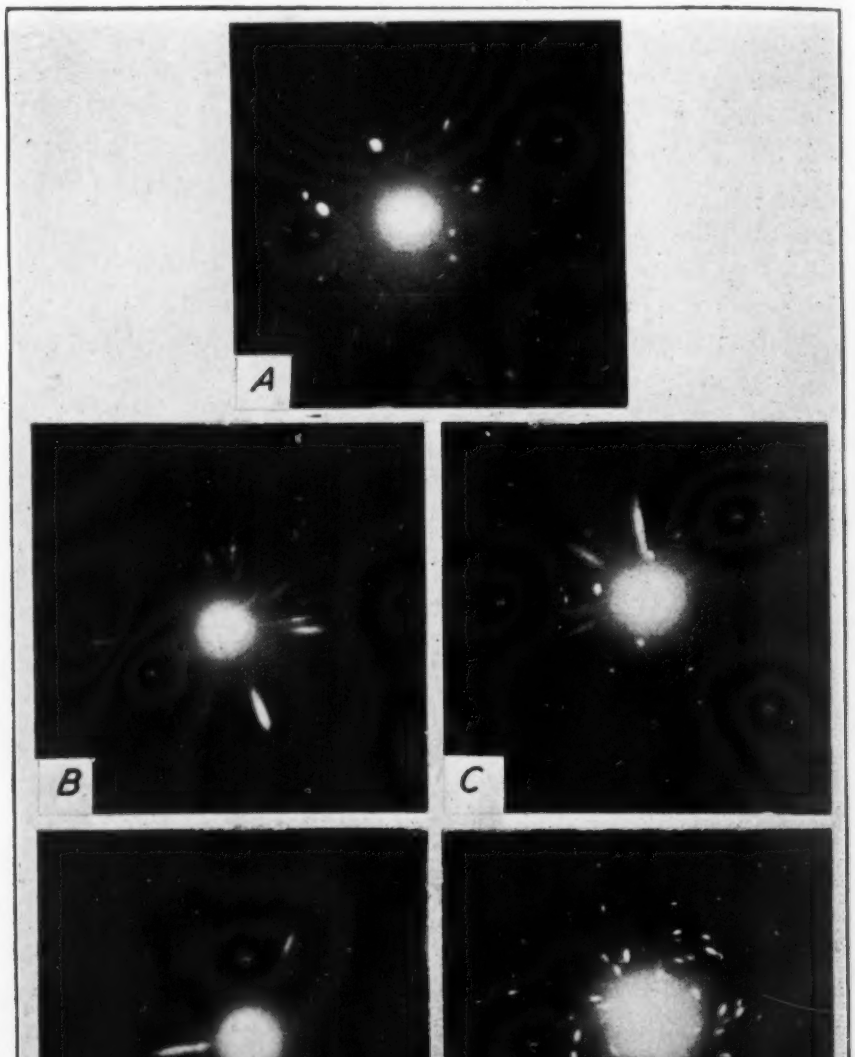


Fig. 6—Pin-Hole Patterns of High-Silicon Transformer Steel Showing Asterism Due to Strain. (a) Annealed Material. (b) Hot-Rolled—1.8 Per Cent Elongation. (c) As in b, but After Annealing. Strain has not been Completely Removed as Evidenced by Residual Asterical Spots. (d) Hot-Rolled —16.1 Per Cent Elongation. (e) As in d, but Annealed. Note Complete Relief of Strain. The Annealing Temperature was the Same as in c.

amounts of strain which in one case (Fig. 6c) was only partially relieved by an experimental annealing and in another case (Fig. 6e)

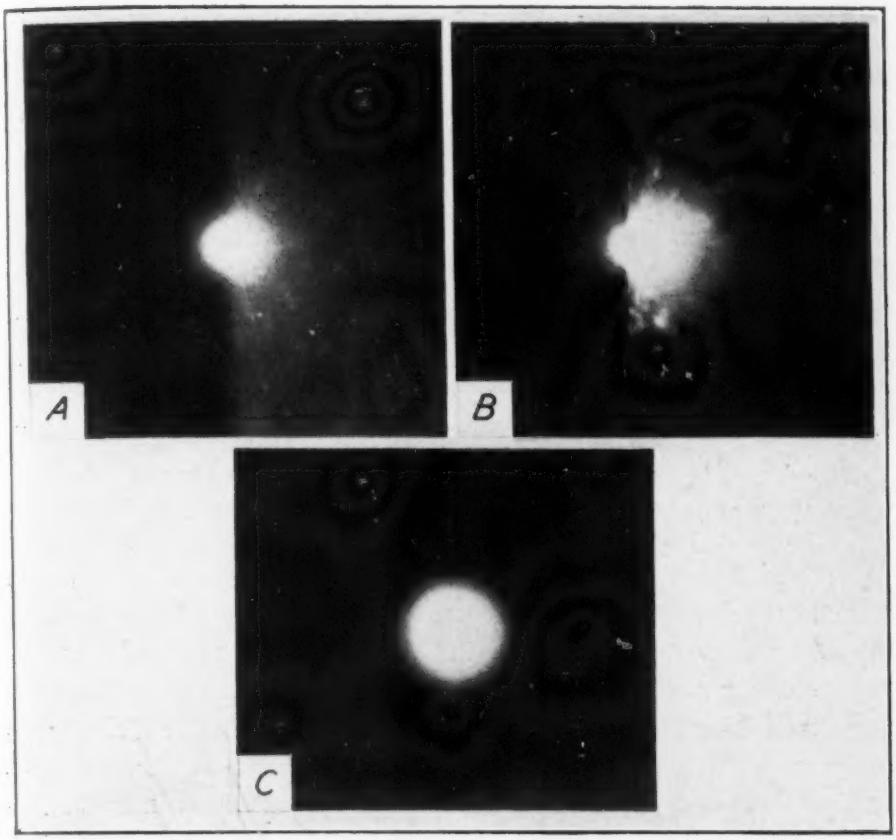


Fig. 7—Effect of Intensifying Screens When not in Pressure Contact on Diffraction Patterns: (a) Intensifying Screen Placed Back of Right Hand Side of Film. (b) Screens in Front and Back of Film on Right Hand Side. (c) Screens in Front and Back Covering Entire Film.

completely relieved by application of the same treatment. Evidence of differences in grain size may also be detected in the patterns. The complete series (not reproduced here in full) revealed an interesting state of affairs as regards the strain-temperature relationship in this material under the conditions of the experiment.

A few experiments were carried out to study the effect of intensifying screens but the results were even less satisfactory than is customary with the use of these screens due, as pointed out by Dr. Davey,[3] to the fact that precautions were not taken to keep them in pressure contact against the photographic films. The results was a blurring of the patterns. These are included, Fig. 7, as evidence of the necessity of taking this precaution. Dr. Davey further

[3]Private communication from Dr. W. P. Davey of Pennsylvania State College to the writer, February 10, 1930.

pointed out that intensifying screens were very sensitive to small quantities of dirt on the surface; that the ratio of intensities (not the order of intensities) of the various spots or lines was altered, and that their intensification is most marked for the high intensities (where intensification is least needed) and least marked for low intensities (where intensification is most needed). The latter point is particularly pertinent to diffraction work where all intensities are relatively extremely weak as compared with, for example, the intensity of the undeviated or "zero" beam. Again, intensification according to the writer's experience, is practically nil at low voltages (30 kilovolts) but somewhat more effective at high voltages (160 kilovolts). In rare instances, such as in orientation studies of the type carried out by Wilson,[4] the greater intensity and subsequent time saving obtained through their use might be warranted. Care should be taken, however, to obtain occasional films without screens to avoid overlooking any points of importance. In most cases, where a complete knowledge of the properties of the materials is desired, the writer believes their use should be avoided.

In some types of work, particularly where the crystal size of the material being studied is small enough to produce "ring" patterns, it may be desirable to use an X-ray beam of known wave length. A beam composed of few wave lengths is usually sought. Such a beam is approximated with tungsten radiation by operating above 70 kilovolts. This is the point at which the K radiation begins to be generated more copiously than the general radiation. Kaye[5] states that the optimum voltage for accomplishing this is about 100 kilovolts. Operation of the tube at this voltage permits one to take advantage of the greater penetrating power afforded by the higher voltage and, at the same time, to avoid short wave length radiation. Use of a filter composed of the same metal as the target is of further assistance in the production of a homogeneous X-ray beam. A filter composed of a metal of slightly lower atomic number would serve still further to purify the beam. The use of tungsten, however, limits the wave lengths to the $K\alpha$ and $K\beta$ components of the spectrum. Kaye states that a tungsten filter, 0.15 millimeter in thickness, removes most of the "general" radiation. With this in view,

[4] "Studies in Metal Crystal Orientation. I. Determination of orientation of metallic single-crystal specimens by high-voltage X-rays," Thomas A. Willson, American Institute of Mining and Metallurgical Engineers, Technical Publication No. 210, Feb. 1929.

[5] G. W. C. Kaye, "X-Rays", 1926, Longmans, Green, & Co., London, New York, p. 252.

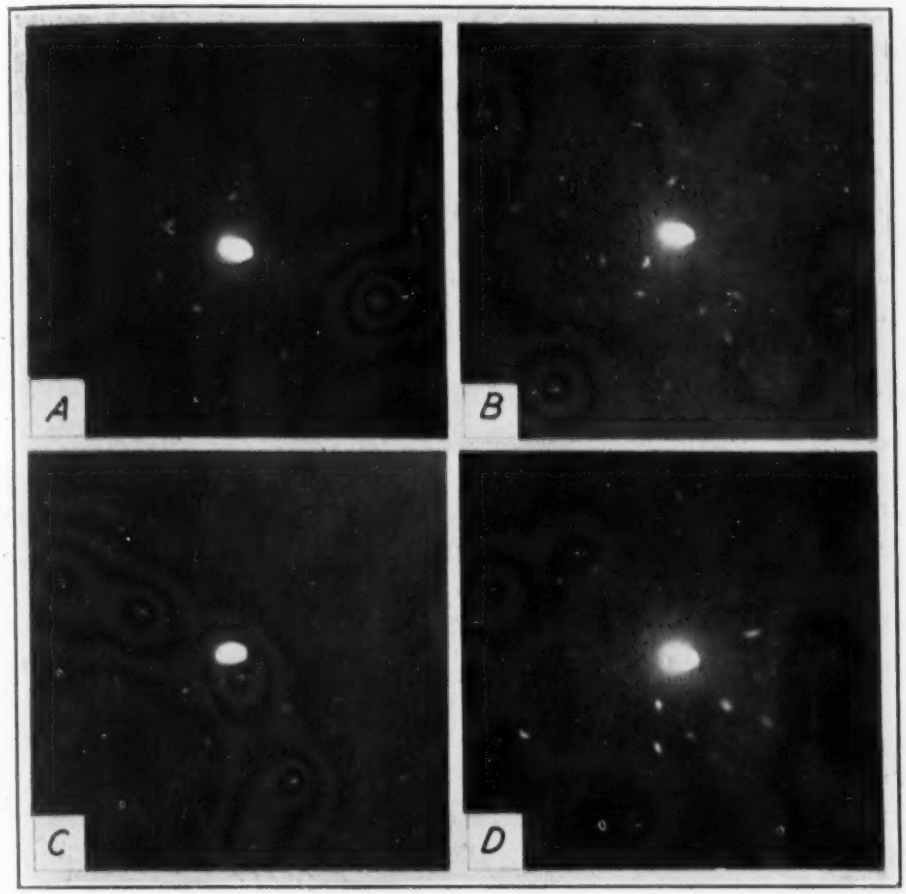


Fig. 8—Comparison of Effect of "Optimum" and Higher Voltage on Diffraction Patterns. (a) Optimum Voltage—100 Kilovolts. (b) Higher Voltage—163 Kilovolts. (c) Optimum Voltage—another specimen. (d) Higher Voltage.

the writer obtained[6] a sheet of tungsten 5 mils in thickness. Unfortunately a diffraction pattern of sufficient intensity was not obtained under the conditions of exposure used, that is: 100 kilovolts, 5 milliamperes, for periods of two hours. This thickness of material is therefore considered to be too great to permit suitable transmission of diffracted rays arising from a crystalline structure under the present conditions. The use of the optimum voltage alone for obtaining a homogeneous X-ray beam was therefore chosen.

The results of tests in which the optimum and a higher voltage were compared are presented in Fig. 8. The voltages used are indicated in the legends. It is rather surprising to note that in the

[6]This tungsten sheet was kindly furnished by the Special Products Section of the General Electric Co.

case of Figs. 8a and 8b ("optimum" and "high"voltage) no appreciable difference in the number of diffraction spots can be detected. This condition was hardly to be expected from X-ray beams differing in number of wave lengths. In another case, however, Figs. 8c and 8d, fewer diffraction spots were obtained with the optimum than with the high voltage. The latter is believed to constitute the anomoly, rather than the former, the reason for which will be mentioned later. Hence it appears that the X-ray beam is but little affected, so far as the wave lengths used to produce the diffraction pattern are concerned, by increasing the voltage above the critical. The fewer spots serving to make up the diffraction pattern reproduced in Fig. 8c, might be attributed to the poorer irradiation of the specimen rather than, for example, to the decrease in the number of available wave lengths. (Similar phenomena have been observed in duplicate exposures of a given group of crystallites under practically identical operating conditions).

The reason for the difference in the degree of irradiation of a given area is unknown to the writer but a few points will be mentioned which might throw a little light on the subject. If we look to some alteration in the character of the X-radiation which precludes any allusion to voltage or other electrical changes, we are confronted with one of two explanations: that there was some change in the character of the electron stream giving rise to the X-rays, or that there was some change in the condition of the surface of the target. In discussing the former possibility we might borrow a line of reasoning from Coolidge[7] which was put forward to explain the observed deviation of the intensity maximum of the X-ray beam from the "heel" of the target. Coolidge stated that, "the cathode ray stream does not remain parallel to the axis of the tube but is electrostatically deflected towards the toe of the target. It may very well be then that the intensity maximum lies in direction perpendicular to the cathode rays at the point of their impact with the target face * * *". Extending this reasoning further and applying it to the case in hand, we might state that the electron stream does not maintain a fixed relation to the axis of the tube under slight variation in operating conditions. The distribution of intensity of the X-ray waves, then, does not remain constant from exposure to exposure. That the focus of the electron

[7]See reference 2.

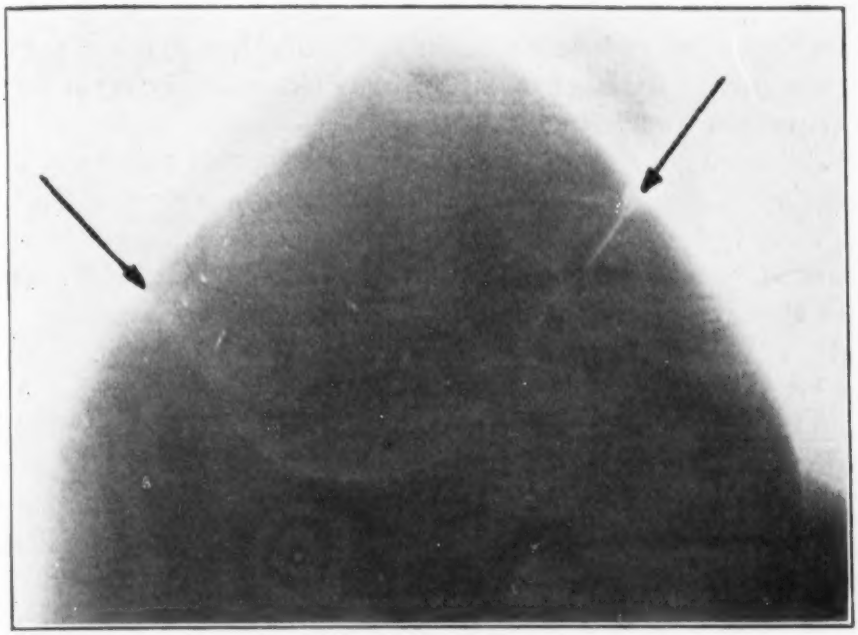


Fig. 9—Radiograph of 5/8-Inch Sheet-Bar Cropping Showing Seam Due to Lapping of Metal. Exposure: 7 Minutes at 160 Kilovolts, 5 Milliamperes Focal Distance 30 Inches, Lead Filter Cassette.

stream remains essentially fixed during a single continuous exposure, however, is believed to be the case in view of some evidence previously obtained. In the course of some radiographic work in which the image of a small object was enlarged to various "magnifications"[8] it was noted that the image of the target of the X-ray tube, obtained simultaneously with that of the specimen, showed a spiral shadow on its face. This was taken to be due to the variation in intensity of a cross-section of the X-ray beam emanating from the target face. The spiral nature was thought to be traceable to the spiral filament of the cathode; that is, the electron stream emanating from the filament was believed to retain the characteristics (in cross-section) of the filament, and that its imprint upon the face of the target would be of a spiral nature also. Hence the intensity of the resultant X-radiation (at 90 degrees) would retain the characteristic of a spiral. The above, as stated at the beginning, is intended to show that the focus of the electron stream is fixed during a given exposure and does not wander appreciably so as to permit variation in irradiation of a given field.

[8] "Note on the Possibility of Enlargement by X-rays", F. Sillers, Jr., *Metals and Alloys*, Vol. 1, No. 5, 1929, p. 225.

The second hypothesis mentioned, that of the pitting of the target, might be tenable. Variation of the X-ray beam through the deflection of the electron stream might conceivably occur due to a shift caused by pitting.

The point of the experiment, however, is that one may resort to higher voltages than the optimum without introducing any significant variables into the diffraction patterns from increase in the number of wave lengths in the beam. Thus we may take advantage of the shorter exposure-time afforded by these higher voltages.

In Fig. 9 is reproduced a radiograph of a cropping from a sheet bar. The effect indicated by arrows is due to a folding over of the metal at the tip as it enters the rolls. While the seam at the surface indicating the extent of the lapping is clearly shown, the region beneath which must extend obliquely under the surface does not register. The void, or oxide layers, are probably too minute to produce any change in intensity over the unseamed part. Conceivably the void might be detected if the X-rays impinged upon it at the proper angle. For example, a fissure encountered in "flaky" steel should be radiographed normal to its narrowest dimension; if it is illuminated at right angles to this direction it is likely to escape detection. Such a plan would not be feasible, however, in the present case.

The radiograph reproduced was one of a series intended to furnish information concerning the usefulness of the radiographic method in the study of sheet steel. At present this field has been little explored and the value of the radiographic method, if any, cannot be appraised. Certain cases, however, suggest themselves to this line of attack. This may be because other lines of attack have not fully elucidated them. In the radiograph in question, no effort was made to shield the edges of the specimen. If these are of interest, however, the specimen is mounted, so to speak, in a plastic substance opaque to X-rays.

It is believed that the films reproduced in this article indicate what may be expected of the instrument described and serve to point out along what lines improvement or modification, necessary for adapting it to other methods of diffraction work, might be desirable. The patterns also indicate that there are certain uses to which the instrument might be put in metallurgical problems. It has been the writer's experience that in several special cases specimens which

appeared very similar in structure under the microscope showed conspicuous differences to the X-ray.

The short period of time required for an exposure with the present arrangement (for the type of material cited) suggests its adoption for the study of processes, or its use as a routine inspection method in a manufacturing process to which it is applicable. Frequently, sufficient time elapses between the various operations to permit the examination of representative samples in order to determine the course that further treatment of the material shall take. If the present method were to be resorted to, four films could be obtained simultaneously, the exposure being in the neighborhood of 1 hour. Occasionally, when lower voltages are used, it becomes necessary to increase this time to, say, 3 hours. In a twenty-four hour day, 96 diffraction patterns could be obtained from coarse-grained material of the type mentioned. It is not supposed, however, that such a great amount of sampling would have to be resorted to under normal plant operating conditions; a dozen films, taken at intervals during a twenty-four hour period, might suffice.

The present instrument is modified from time to time in the event that some new type of crystallographic information is desired. On one occasion it was thought desirable to employ a slit-system to replace the present pin-hole arrangement in the examination of some fine-grained material and the diffractometers were modified accordingly. When such a line slit is used, a semi-circular film-holder can be substituted at any time for the flat film holders shown in Fig. 4. This would permit the accurate measurement of interplanar spacings in the usual way. It is quite possible that the instrument could be adapted to other established methods of crystal analysis if the necessary accessories were secured.

RADIOGRAPHY BY THE USE OF GAMMA RAYS

By Robert F. Mehl, Gilbert E. Doan, and Charles S. Barrett

Abstract

The possible uses of γ-rays for detection of defects in metallurgical materials have been made the subject of a preliminary investigation. The physics of γ-rays from the standpoint of radiography have been explored, and radiographic experiments performed. It has been found that, given a sufficient source of γ-rays, satisfactory radiographs may be taken of steel objects the thickness of which far exceeds that now possible by means of X-rays. Radiographs have been taken with some degree of success through ten inches of steel. These results are discussed, with notes upon possible practical use of the method.

Introduction

THE use of X-rays for taking shadow pictures of metallurgical and other materials, has, during the last fifteen years, been made the subject of many articles in various scientific journals. It is now admitted that radiography by the use of X-rays is very serviceable in the detection and location of defects characterized by density differences, and accordingly industry is increasingly adopting the method.

There is, however, another type of radiation which holds potential value for radiography, namely, the γ-rays emitted by radioactive substances. They are rays much more penetrative than ordinary X-rays, and would appear to possess certain other advantages for radiographic use. Since X-rays seem limited for practical use to sections 3½ to 4 inches at most in thickness, and since industry, owing to the development of larger structures and of high temperature and high pressure processes, frequently desires information on parts thicker than this, any radiographic method capable of inspecting thicker sections should be of interest.

Contribution from the Division of Physical Metallurgy, Naval Research Laboratory, Washington, D. C. Published by permission of the Navy Department, Washington, D. C.

A paper presented before the Twelfth Annual Convention of the society in Chicago, September 22 to 26, 1930. Of the authors, Robert F. Mehl is superintendent, Division of Physical Metallurgy, Naval Research Laboratory, Washington, D. C.; Gilbert E. Doan, assistant professor of metallurgy, Lehigh University, Bethlehem, Pa.; and Charles S. Barrett, assistant physicist, Division of Physical Metallurgy, Naval Research Laboratory, Washington, D. C.

The literature is surprisingly deficient in information upon the radiography of opaque objects by γ-rays, except for statements that radiographs of the human body obtained by the use of γ-rays do not show details as clearly as do X-rays, that is, that the contrast is not so great. As we shall see, this is because of the lower absorption or greater penetrability of the γ-ray. Years ago, Rutherford,[1] noted that gamma rays from 30 mg. of radium could be detected with an electroscope after passing through 30 cm. of iron. Thin strips of metal have been radiated by γ-rays and the transmitted ray observed upon a fluorescent screen. But so far as we have been able to discover there has been no attempt to use γ-rays for the inspection of opaque bodies except that reported from Russia.[2] According to the meager information available it was found possible to locate flaws in steel castings, detecting the emergent beam by an electroscope or an ionization chamber and recording the intensity of the beam automatically. No details of this work have ever been made public, so far as the present authors are aware.

It may be said, therefore, that but little has been done on the use of γ-rays for radiography. The present investigation is an attempt, in a preliminary way, to evaluate the use of γ-rays for the detection and location of defects in metallurgical materials.[3]

The Physics of γ-rays

In order that the ensuing account may be made clear it is thought advisable to comment briefly upon certain of the physical characteristics of γ-rays. Since the source of γ-rays used was radium emanation, the most suitable source, these notes will be restricted to γ-rays as obtained from radium emanation.

Possibly the most noteworthy fact about the radioactive elements is that they transform or decompose to chemically and physically different elements. These transformations are accompanied by radiation, of which there are three types, the α-rays, which are known to be helium atoms with a double positive charge, β-rays, which are electrons, and γ-rays, which are light waves of very short wave-

[1] E. Rutherford, "Radioactive Substances and Their Radiations", Cambridge University Press, 1913, p. 257.

[2] As published by Science News Service, No. 361E, Sheet 1, Feb. 24, 1928. It has been impossible to obtain by correspondence information additional to that appearing in the Science News Service report.

[3] The experiments described in the following were performed during the summer of 1929.

length. The first product in the decomposition of radium is radium emanation, or radon. It is a chemically inert gas, over a hundred times heavier than hydrogen. This gas, radon, in turn decomposes and forms successively Radium A, Radium B, Radium C, Radium D, etc., with the final product lead.

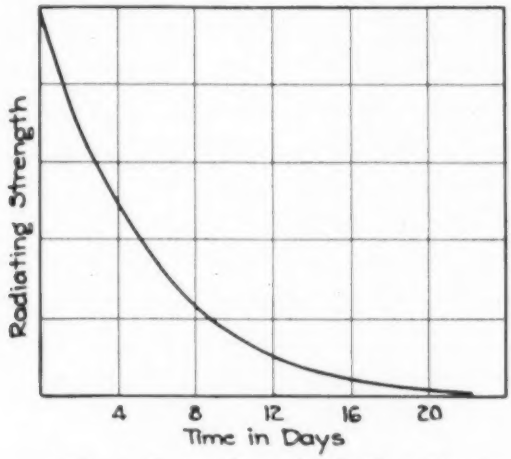


Fig. 1—Decay Curve for Radium Emanation.

Each of these radioactive elements is characterized by a certain rate of decomposition. If the amount present be evaluated (in the usual way) by the strength of the radiation emitted the rate of decay may be given as

$$\frac{I}{I_o} = e^{-\lambda t}$$

where I is the intensity measured after time t, I_o the original intensity, and λ is the fraction of atoms present which transforms in time t. This is a general law for all radioactive substances. Accordingly, the strength of all radioactive substances decreases logarithmically with time. (Fig. 1). Since it is impossible to speak of the life of the emanation (for it is infinite), it is customary to speak of the half-life period, that is, the period in which the radioactive strength decreases to half its original value.

The half-life of radium is about 1600 years. It may, therefore, over a short period be considered as unchanging. Granted this it may be seen that a given quantity of radium produces a constant quantity of radium emanation each second, which in turn decomposes to Radium A, and so on. With a constant rate of production of radium emanation the amount of radium emanation will build up

until the rate of decay of the emanation exactly balances the rate of its formation. At this point the radium and its emanation are said to be in equilibrium.

The transformation of radium to radium emanation is accompanied only by an emission of α-rays. So far as γ-rays are concerned the radioactive properties of radium in equilibrium with its products are therefore possessed completely by the radium emanation, and for practical purposes it is better to use the emanation.

In medical practice radium emanation is pumped from the parent radium salt (usually in solution) and condensed from the gases with which it is mixed by liquid air. Each gram of radium in the equilibrium state furnishes 0.6 cubic millimeters of gas. This condensed gas is pumped into small glass capsules ordinarily held in small brass containers. The chief source of γ-rays seems to be Radium C, into which radium emanation decomposes. This series of transformations with the radiations which accompany them and with the half-life periods, is indicated by the following:[4]

$$\underset{1580\ \text{yrs.}}{\text{Ra}} \xrightarrow{\alpha} \underset{3.85\ \text{days}}{\text{Ra Eman.}} \xrightarrow{\alpha} \underset{3.05\ \text{min.}}{\text{RaA}} \xrightarrow{\alpha} \underset{26.8\ \text{min.}}{\text{RaB}} \xrightarrow{\beta,\gamma} \underset{19.5\ \text{min.}}{\text{RaC}} \xrightarrow{\beta,\gamma} \left\{ \begin{array}{l} \underset{10^{-6}\ \text{sec.}}{\text{RaC'}} \xrightarrow{\alpha} \\ \underset{1.32\ \text{min.}}{\text{RaC''}} \xrightarrow{\beta,\gamma} \end{array} \right\} \underset{1.32\ \text{min.}}{\text{RaD}}$$

As freshly pumped off the emanation reaches its maximum radiating intensity within a few hours, the time necessary for the production of the maximum amount of RaC. Thereafter the strength decreases logarithmically according to Fig. 1, the half-life period being 3.9 days.

The strength of the emanation is measured in millicuries, which is the strength of 1 milligram of radium in equilibrium with its products as measured by an electroscope receiving the γ-rays only. In medical practice it is customary to pump off the emanation each day. With this procedure one gram of radium will furnish 160 millicuries of the emanation, of which about 150 millicuries is recovered by a skilled operator. Accumulating this emanation day by day will build up a permanent stock of emanation with a total strength between 800 and 900 millicuries available for radiographic purposes.

Wave-length of γ-rays. Approximations have been made indicating[5] that the γ-ray spectrum lies somewhere in the range 4.100 to

[4] See, for example: A. Sommerfeld, "Atomic Structure and Spectral Lines," Methuen & Co., Ltd., London, 1923, page 47.

[5] Meyer and Schweidler, "Radioaktivität," Teubener, Berlin, 1927, p. 148.

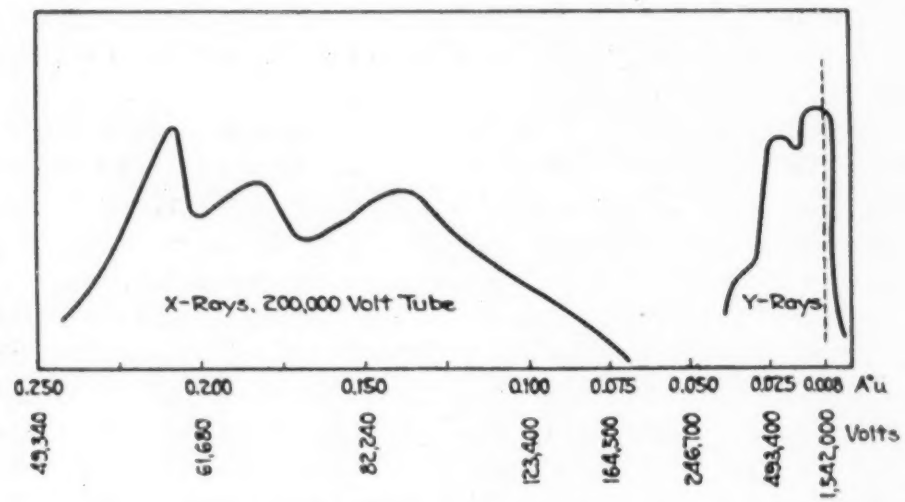


Fig. 2—Curves Showing Intensities at Various Wave-Lengths for X-rays (Terril and Pine) and for γ-rays (Skobelzyn). Dotted Line Represents the Mean Value for γ-rays Given by Ellis and Wooster.

0.005 Å.U. The distribution of intensity in the γ-ray spectrum has recently been studied by a number of workers.[6]

The relative intensities found by Skobelzyn for the various wave-lengths in the γ-ray spectrum are shown in Fig. 2.[7]

The measurements made by Ellis and Wooster suggest a mean wave-length for the γ-ray spectrum of 0.008 Å.U., corresponding to a voltage of 1,500,000 volts as indicated in Fig. 2, somewhat shorter than the approximate mean wave-length to be derived from Skobelzyn's curve. On Fig. 2 is also represented the relative intensities found by Terrill and Pine[8] for the various wave-lengths in the X-ray spectrum as obtained from a 200,000-volt X-ray tube with

[6]D. Skobelzyn, Z. Phys. Vol. 43, 1927, p. 368; K. W. F. Kohlrausch, Phys. Zeit. Vol. 28, 1927, p. 2; J. Thibaud, Ann. d. phys. Vol. 5, 1925, p. 119; C. Ellis and W. Wooster, Proc. Camb. Phil. Soc. Vol. 22, 1925, p. 853, Vol. 23, 1925-27, p. 717-729.

[7]It seems likely that the γ-ray spectrum, originating from an atomic disintegration, is not a "white" or continuous spectrum but a spectrum of discrete lines. As the ray emerges from the atom, however, it doubtless suffers a modification in wave-length owing to interaction with the radioactive substance itself and with any filter in its path, converting the separate rays through Compton scattering into a continuous spectrum. Skobelzyn determined the relative intensities of the different wave-lengths by the number of recoil electrons of given velocity (corresponding to a given wave-length of γ-ray) in a Wilson cloud chamber. His curve is "ironed out" in Fig. 2 to give a smoother curve than he observed and Fig. 2 therefore makes the intensity distribution appear more equable than it probably is. There is still some question of where the limits of the γ-ray spectrum lie. There is evidence of rays at wave-lengths greater than 0.040 Å.U., and some suggestion of rays around 0.002 Å.U. The limits of the γ-ray spectrum are therefore not defined in Fig. 2. In addition, Skobelzyn's determination of distribution of intensities rests upon only 160 observed recoil electron paths, a number insufficient to define the intensity distribution curve with high accuracy. The curve given in Fig. 2 may, however, be taken as approximately correct. The data from which Fig. 2 was plotted refer to γ-rays filtered through 3 mm. of lead.

[8]H. M. Terrill and Mary Pine, Journal of Cancer Research, Vol. 8, 1924, p. 71.

a tungsten target after filtering through 1 mm. of copper. It is to be noted that the γ-ray curve in Fig. 2 applies to γ-rays after filtering through 3 mm. of lead whereas the X-ray curve was taken with 1 mm. copper filter; this difference in filters exerts some distorting effect upon the true distribution curves, but for the present purpose the curves given in Fig. 2 may be taken as a nearly true index of the distribution of wave-lengths in the two spectra.

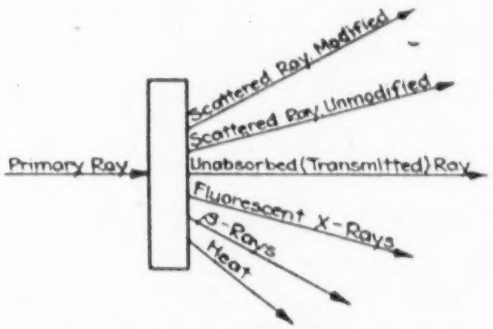


Fig. 3—Changes Suffered by Radiation in Penetrating Matter.

The areas under the intensity curves in Fig. 2 are entirely dependent upon the intensity of the source, whether it be an X-ray bulb or a tube of radium emanation. In Fig. 2 the two curves are drawn simply to give approximately equal heights. It is, of course, the much shorter wave-length of the γ-ray as indicated in Fig. 2 which accounts for the much lower absorption of the γ-rays in radiography.

Absorption and Scattering. The γ-ray beam issuing from a bulb of radium emanation has approximately the distribution of wave-lengths indicated in Fig. 2 (after, as noted, passing through 3 mm. of lead, which absorbs the softer rays preferentially). As this ray passes through a metal object it suffers a number of changes. The emergent ray is complex, as indicated in Fig. 3.

The emergent γ-ray beam is weaker than the direct beam because of loss of intensity through simple absorption (converted into heat) and through scattering. This emergent intensity may be represented by an equation.

$$I = I_o e^{-\mu x}$$

where I_o is the initial intensity (assumed to be a simple wave-length), x the thickness of the absorbing medium, and μ the total absorption coefficient.

The total absorption coefficient μ is actually affected by two agencies, namely, true absorption and scattering. Neither X-rays, and γ-rays from a bulb of radium emanation, are homogeneous in wave-length (Fig. 2). Accordingly the first absorbing layers remove a large fraction of the less penetrating rays, so that only the more penetrating or "hard" rays reach the final layers. For such non-homogeneous beams the effective value of μ is much greater for the rays which enter the absorbing object than for those that leave it. According to Kohlrausch[9] the total absorption coefficient for γ-rays may be represented by two absorption coefficients, μ_1, and μ_2, where

$$I = I_0 \left(e^{-\mu_1 x} + 0.75\, e^{-\mu_2 x} \right)$$

For iron $\mu_1 = 0.356$ and $\mu_2 = 0.632$ cm.$^{-1}$, with an average value in the neighborhood of 0.5 cm.$^{-1}$.

Taking 0.15 Å.U. as the effective wave-length of X-rays from a 200,000-volt tube (Fig. 2) the corresponding coefficient for iron is 4.5 cm.$^{-1}$ These coefficients thus indicate the greater penetrability of the γ-rays.

It has been stated that the total absorption coefficient represents the sum of a true absorption coefficient and a scattering coefficient. The scattered ray is of considerable importance to radiography, since it commonly fogs the registering film. To combat the scattering various screening devices have been introduced, such as simple lead shields and the more complicated Potter-Bucky diaphragm.[10]

The scattering of γ-rays differs from that of X-rays owing to the shorter wave-length. The difference is in three categories: (a) the fraction of the beam scattered (the magnitude of the scattering coefficient), (b) the angular distribution of the scattered ray, and (c) the modification of the wave-length upon scattering[11] (Compton effect).

(a) It is difficult to make any definite statement concerning the ratios of the true absorption and scattering coefficients for X-rays and γ-rays, except that both these coefficients are smaller for γ-rays than X-rays. There is a lack of data from experiments duplicating radiographic conditions.

[9] Meyer and Schweidler, "Radioaktivität," Teubener, Berlin, 1927, p. 644.

[10] See, among many publications, that of the Eastman Kodak Company, entitled: "X-rays in Industry," 1929.

[11] Fluorescent radiation in both the X-ray and γ-ray technique is absorbed before reaching the film and need not be considered here. The ejected photoelectrons suffer a similar fate.

(b) The angular distribution of intensity is of peculiar interest in radiography. Both the modified and the unmodified ray are distributed more in the direction of the primary beam in the case of the shorter wave-lengths than in the case of the longer. Under radiographic conditions the modified rays predominate over the unmodified at practically all scattering angles, so that a curve of angular

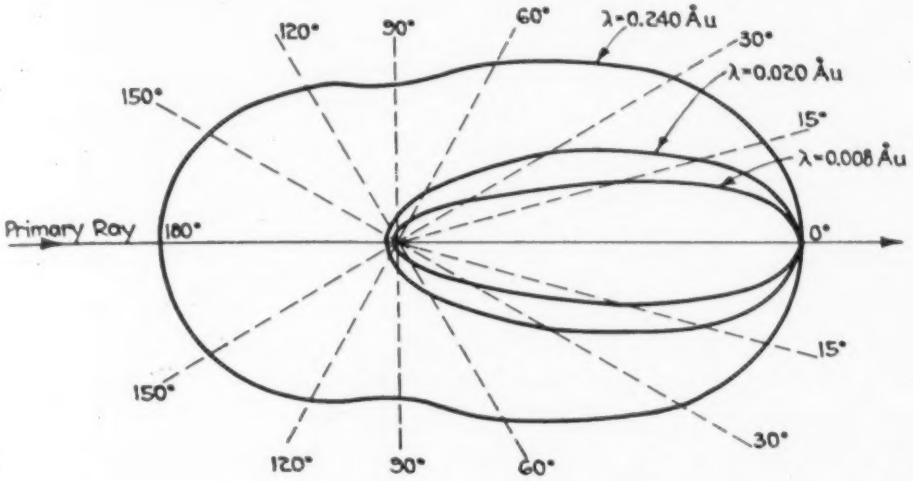


Fig. 4—Distribution of Intensity of Modified Rays Scattered at Various Angles, with Primary Rays of Wave-Length 0.240 Å. U. (X-Rays), 0.020 and 0.008 Å. U. (γ-rays).

distribution for the former gives approximately the distribution of the total scattering. Fig. 4 gives a series of such curves,[12] and indicates that the distribution of the scattered ray should be less detrimental to the production of good γ-ray radiographs than to good X-ray radiographs.

(c) The scattered γ-ray beam suffers a profound modification through the operation of the Compton effect, and in this respect differs greatly from X-rays. The Compton scattering is an increase in the wave-length of the primary beam, the amount of increase dependent upon the angle the scattered beam forms with the primary beam but independent of the original wave-length. A beam scattered at an angle 90 degrees to the primary beam suffers an increase in wave-length of 0.024 Å.U. Such an increase will make only relatively little difference in the scattered X-ray wave-lengths (since the effective wave-length of the primary beam from a 200,000-volt tube is about 0.15 Å.U.) but will make a great difference in the scat-

[12]K. W. F. Kohlrausch, "Handbuch der Experimentalphysik," Vol. XV, p. 132.

tered γ-ray wave-lengths (which are principally between 0.030 and 0.004 A.U.) dependent upon the angle of scattering.

The scattered γ-ray beam will therefore have a much greater wave-length at large angles and a proportionately much greater absorption coefficient. This will, in effect, so far as radiography is concerned, narrow the angular distribution of the scattered γ-ray beam, since the beam scattered at the greater angles will be more rapidly absorbed by the specimen.

These various considerations indicate that scattering from γ-rays should not be a greatly harmful influence in γ-ray radiography, and the degree of success attained in the experiments soon to be described bears testimony to the correctness of this viewpoint.

Photographic Exposure Characteristics. A great deal has been written about photographic exposure characteristics for radiography by the use of X-rays. The information desired is simply the shortest exposure time for a given thickness of metal necessary to reveal upon the developed photographic film the density differences—defects—occurring in the object inspected.

There is a fairly wide latitude in the choice of exposure times for different thicknesses, dependent upon the observer's taste in film density and upon what deviations from the simplest technique are used, such as enhancing screens, special developing methods, and special film inspecting methods.

To eliminate the fancies of the observer exposure curves may be given to represent the proper exposures for a given degree of absolute blackening of the film. The absolute blackening is determined by photometric measurements in which the intensity of the light is measured thermoelectrically. The degree of blackening, or density, is then given by the expression

$$D = \log_e \frac{I_o}{I} = 2.3 \log_{10} \frac{I_o}{I}$$

where D = density, I_o = intensity of primary beam, and I = intensity of transmitted beam.

A density of D = 1.15 is sometimes taken as a satisfactory degree of blackening for radiographic purposes.[13] This density refers to the increase in density above that of the unexposed but developed film.

[13]In Briggsian logarithms a density of 0.5.

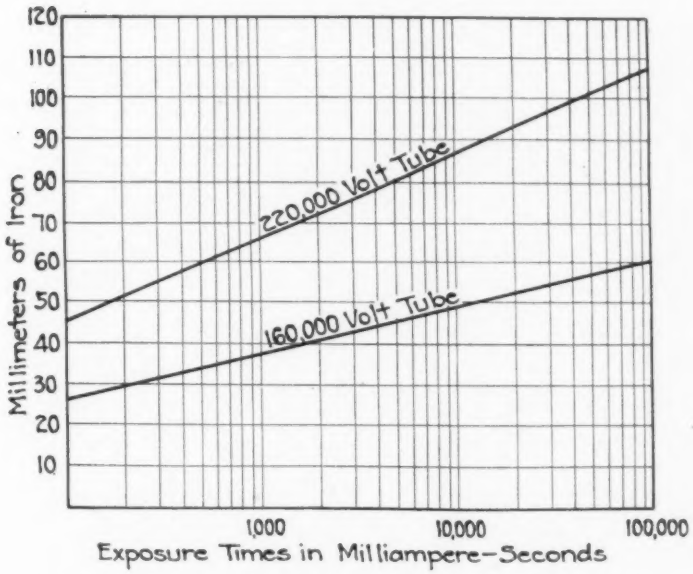


Fig. 5—Exposure Curves for X-rays; for a Distance of 16 Inches from Source and with the Use of Two Intensifying Screens.

Fig. 5 gives such exposure curves for X-ray tubes operating with a tungsten target[14] at the indicated voltages (D. C.). There is at present no exposure curve available for γ-rays.[15] Neglecting scattering and nonhomogeneity of wave-length, an exposure curve should have a slope inversely proportional to the linear absorption coefficient of the ray used (when plotted in the usual way, thickness against the logarithm of milliampere-seconds). Mathematically

$$x = \frac{1}{\mu} \log_e kt$$

where x is the thickness, t is the time of exposure required for a given blackening (for a constant tube milliamperage in the case of X-rays or for a constant millicurie strength in the case of γ-rays), μ is the linear absorption coefficient as before, and k is a constant depending upon experimental conditions. A change in μ is, therefore, much more effective in changing x than a change in t, since the logarithm of t changes very slowly with t. In other words, to increase the thickness range in radiography, a decrease in wave-

[14]Adapted from "Der gegenwärtige Stand der Röntgendurchstrahlung von Metallen," Siemens-Reiniger-Veifa, Berlin, 1928.

[15]Some attempts were made to procure such curves by a technique which later proved inadmissible. It is hoped shortly to obtain exposure curves to be used in practical γ-ray radiography. The radiographs reproduced in this paper are not of uniform density, nor are they all of the lowest practicable density, so that the exposure times cannot be taken as a guide except in a rough way.

length is much more effective than an increase in time. To illustrate: Consider the difference in exposure time for 3 inches and 6 inches of iron. From Fig. 5 the exposure time for 3 inches with a 220,000-volt X-ray tube is 3000 milliampere-seconds (50 milliampere-minutes), requiring an exposure time of about 10 minutes at a milliamperage of 5. Extrapolating the exposure curve for a 220,000-volt tube to 6 inches, the exposure time becomes approximately 15,000,000 milliampere-seconds, at a milliamperage of 5, requiring an exposure time of 50,000 minutes. The ratio of these times is

$$\left(\frac{t_{6\text{ inches}}}{t_{3\text{ inches}}}\right)_{\text{X-rays}} = 5{,}000$$

For γ-rays, assuming $\mu = 0.5$, an approximate value for the above ratio may be obtained. The equation

$$x = \frac{1}{\mu} \log_e kt$$

may, for two different thicknesses, be written:

$$x_1 - x_2 = \frac{1}{\mu} \log_e \frac{kt_1}{kt_2} = \frac{1}{\mu} \log_e \frac{t_1}{t_2}$$

substituting 6 inches for x_1 and 3 inches for x_2 the time ratio becomes

$$\left(\frac{t_{6\text{ inches}}}{t_{3\text{ inches}}}\right)_{\gamma\text{-rays}} = 45$$

where the similar time ratio for X-rays from a 220,000-volt tube as given above is roughly one hundred times as great.

Thus, though the requisite exposure time for a 6-inch section with X-rays becomes unconscionable, satisfactory radiographs of even thicker sections may be taken with γ-ray sources now available within reasonable time, as we shall see in the experimental part of the present paper.

The lower absorption of the γ-ray may be shown by another example. Taking 0.5 as the linear absorption coefficient of γ-rays, the intensity of the primary γ-ray beam will be decreased to about $\frac{1}{3}$ by one inch of iron, whereas X-rays, with a linear absorption coefficient of 4.5 (a wave-length of 0.15 Å. U.) would be reduced to about 1/100,000 of the original intensity by an equal thickness of iron.

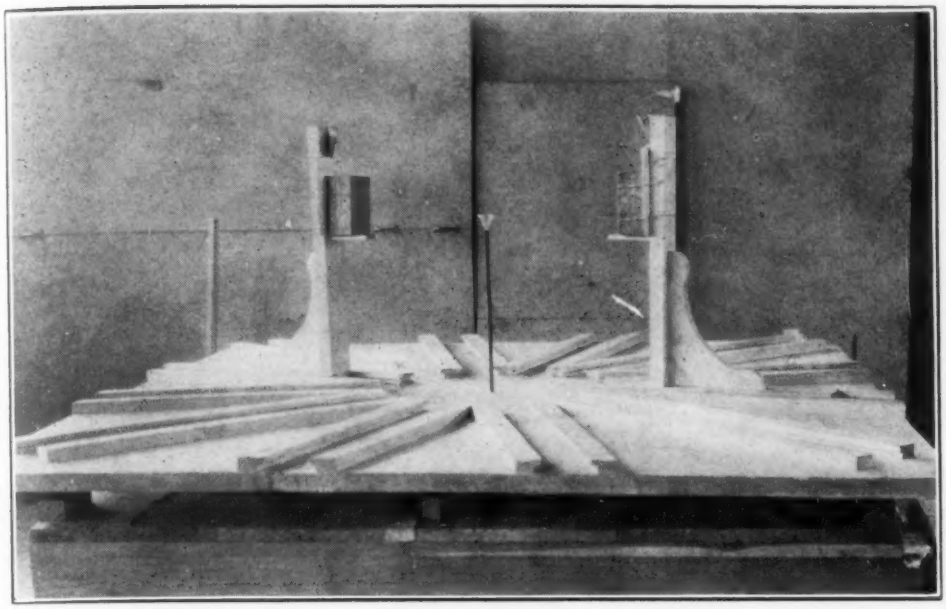


Fig. 6—Table Used in γ-ray Radiography. Funnel Holding Radium Emanation Shown in Center.

In passing it probably should be remarked that the inverse square relation between intensity and distance applies to γ-rays as well as to X-rays. In neither case is there an appreciable absorption in air.

Experimental

Methods of Recording the Emergent γ-ray Beam. There are three possible methods of performing this task: (a) by means of the ionizing effects of the ray, (b) visually, upon a fluorescent screen, (c) by means of a photographic film or plate.

All three of these methods have been tried. The success reported from Russia seemed to warrant an experimental trial of (a).

(a) Owing to the simplicity and efficacy of the photographic method no very thorough study of the ionization method was made, though it is believed that the experiments performed warrant the conclusions drawn.

A cylindrical ionization chamber was constructed, 3 inches in diameter and 12 inches long, lined with lead and provided with twelve partitions of lead-coated brass sheet, six of which were connected to a central rod and six to the outer casing.[16] The central rod was

[16]Similar in construction to that used by A. H. Compton, *Phil. Mag.* Vol. 41, 1921, p. 749.

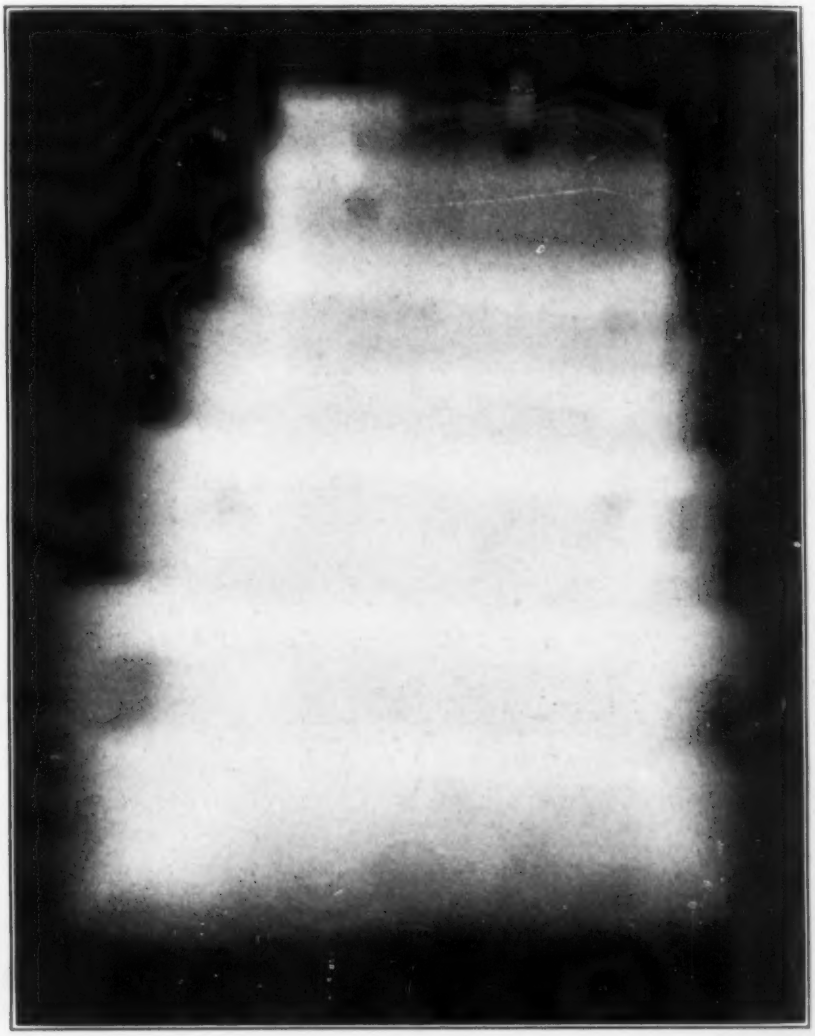


Fig. 7—An Echelon of Steel Plates, as Illustrated in Fig. 8. (Film No. 1)

Distance, source to film: 6 inches
Intensifying screen: None
Time of exposure: 25 minutes
Mean strength of emanation: 179 millicuries (hereinafter simply m.c.) in one bulb

Results: Considerable detail on original negative lost in Fig. 7. Original exposure just sufficient to bring out all details, though enhancing screens would have decreased the exposure time. The spreading on the film was caused, of course, by the closeness of the object to the source.

insulated with quartz and connected to a vacuum-tube amplifier. The outer case was charged to 300 volts. In order to increase the ionization current the chamber was filled with methyl bromide at atmospheric pressure.

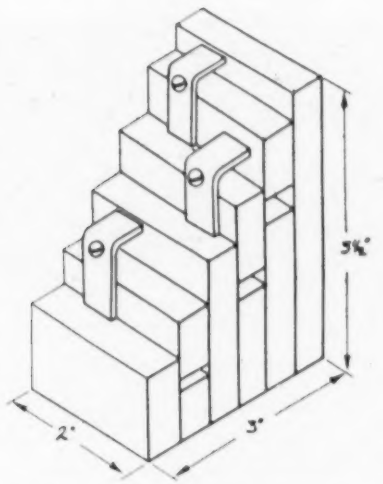


Fig. 8.—Showing Built-Up Specimen Radiographed in Fig. 7.

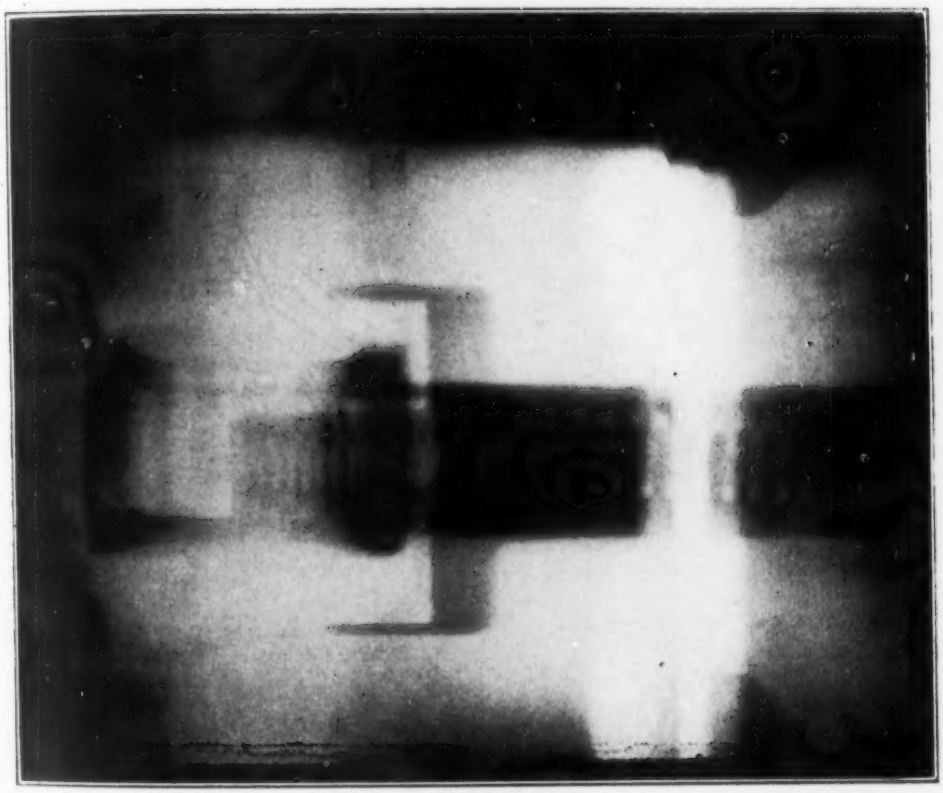


Fig. 9—A Tool Rest From a Lathe. Overall Length of Section Photographed, 8¼ Inches; Height, 6½ Inches. (Film No. 2)

Distance, source to film:	26 inches
Intensifying screen:	None
Time of exposure:	12 hours
Mean strength of emanation:	181 m.c. in one bulb

Results: Film badly over-exposed. With intensifying screens only a small fraction of the exposure time noted would have been necessary. A high degree of detail brought out on the original film.

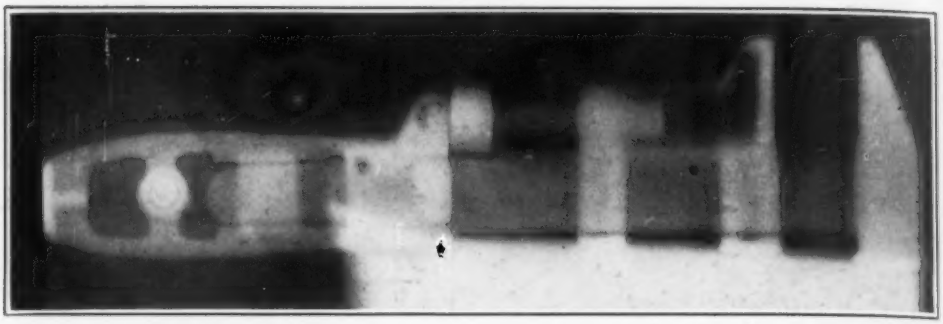


Fig. 10—Wrench, 8½ Inches Long. (Film No. 3)

Distance, source to film:	66½ inches
Intensifying screen:	None
Time of exposure:	10½ hours
Mean strength of emanation:	2181 m.c. in six bulbs

Results: All details shown very clearly. Film somewhat over-exposed. Distance from source very great. With a shorter distance and by the use of intensifying screens, a shorter time and a weaker source would have sufficed. Note increase in sharpness of all details in films No. 1 to No. 3 with increase in distance from source to film, as to be expected. The lack of halo around the edge of the specimen indicates that the usual precautions taken in X-ray radiography to prevent halo are unnecessary when radiographing thin specimens by γ-rays.



Fig. 11—Faulty Weld, Two ½-Inch Steel Plates, Lap-Welded. (Film No. 4)

Distance, source to film:	30 inches
Intensifying screen:	None
Time of exposure:	12 hours
Mean strength of emanation:	217 m.c. in one bulb

Results: Lack of bond clearly shown (indicated by arrow in Fig. 11). Fig. 11 not nearly so good as original film. Exposure excessive. Time of exposure could have been much less, especially by use of Patterson screens.

The vacuum-tube amplifier used was designed after that described by Eglin[17] and was capable of measuring currents of the

[17] J. M. Eglin, Phys. Rev. Vol. 33, 1929, p. 113A.



Fig. 12—Faulty Weld, butt-welded. (Film No. 5).

Distance, source to film:	30 inches
Intensifying screen:	None
Time of exposure:	12 hours
Mean strength of emanation:	179 m.c. in one bulb

Results: Imperfections clearly shown.

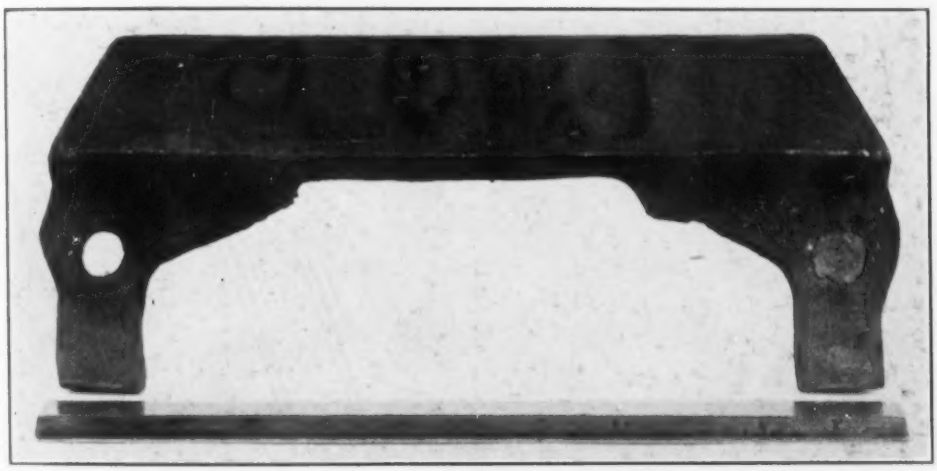


Fig. 13—Photograph of Steel Casting Radiographed in Figs. 14 and 15.

order of 10^{-13} to 10^{-14}. The most satisfactory tubes tried were the English Marconi type D. E. Q.[18] Readings were taken with a Leeds and Northrup galvanometer.[19]

A lead block with a longitudinal hole was placed between the

[18]Amplification factor 20; impedance 100,000 ohms.

[19]Sensitivity 0.7 mm. per microvolt; 46 ohms resistance; 100 to 500 ohms shunt.

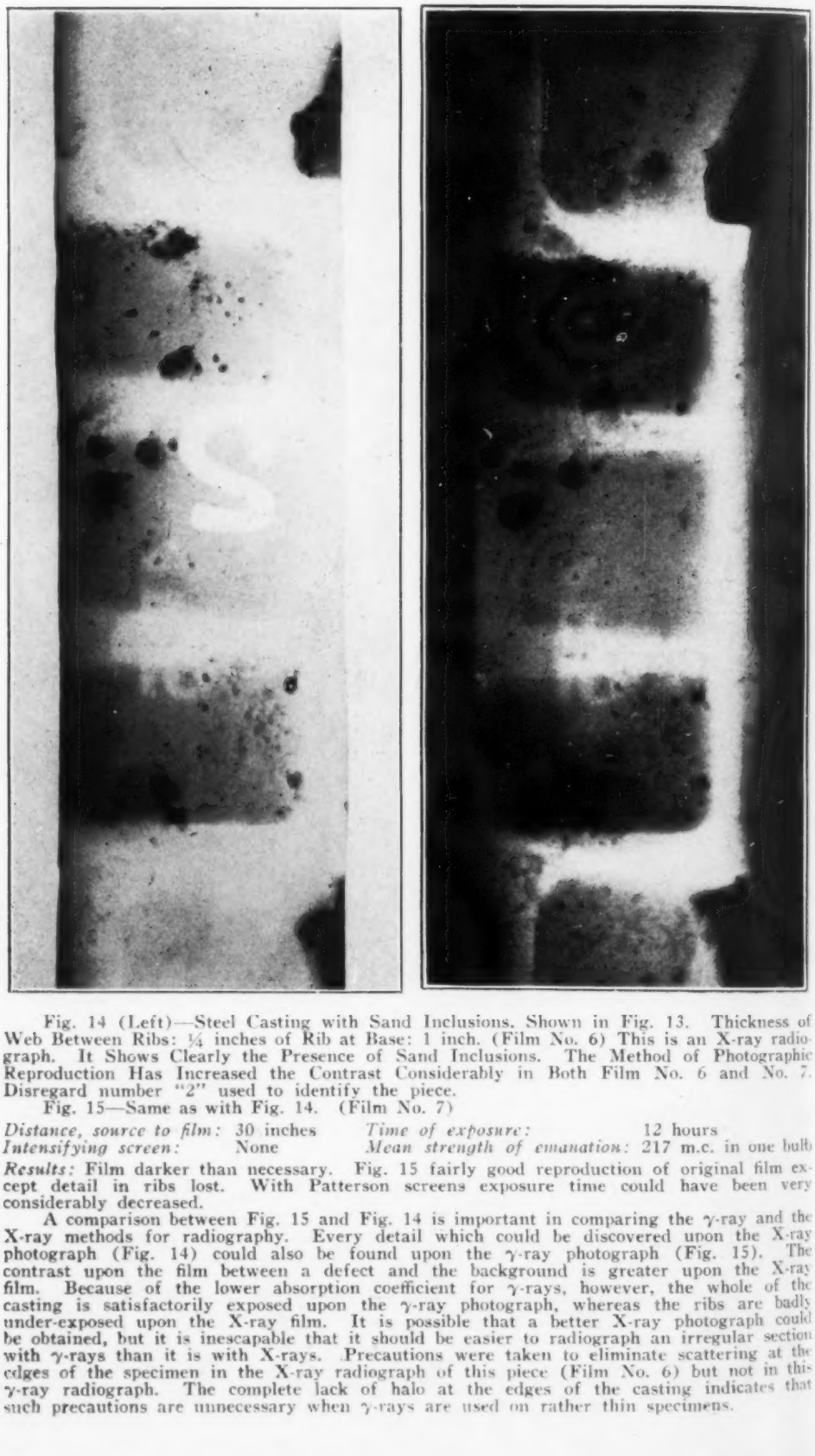


Fig. 14 (Left)—Steel Casting with Sand Inclusions. Shown in Fig. 13. Thickness of Web Between Ribs: ¼ inches of Rib at Base: 1 inch. (Film No. 6) This is an X-ray radiograph. It Shows Clearly the Presence of Sand Inclusions. The Method of Photographic Reproduction Has Increased the Contrast Considerably in Both Film No. 6 and No. 7. Disregard number "2" used to identify the piece.

Fig. 15—Same as with Fig. 14. (Film No. 7)

Distance, source to film: 30 inches *Time of exposure:* 12 hours
Intensifying screen: None *Mean strength of emanation:* 217 m.c. in one bulb

Results: Film darker than necessary. Fig. 15 fairly good reproduction of original film except detail in ribs lost. With Patterson screens exposure time could have been very considerably decreased.

A comparison between Fig. 15 and Fig. 14 is important in comparing the γ-ray and the X-ray methods for radiography. Every detail which could be discovered upon the X-ray photograph (Fig. 14) could also be found upon the γ-ray photograph (Fig. 15). The contrast upon the film between a defect and the background is greater upon the X-ray film. Because of the lower absorption coefficient for γ-rays, however, the whole of the casting is satisfactorily exposed upon the γ-ray photograph, whereas the ribs are badly under-exposed upon the X-ray film. It is possible that a better X-ray photograph could be obtained, but it is inescapable that it should be easier to radiograph an irregular section with γ-rays than it is with X-rays. Precautions were taken to eliminate scattering at the edges of the specimen in the X-ray radiograph of this piece (Film No. 6) but not in this γ-ray radiograph. The complete lack of halo at the edges of the casting indicates that such precautions are unnecessary when γ-rays are used on rather thin specimens.

γ-ray source and the ionization chamber, and served to define a beam half an inch in diameter. Steel blocks of different thicknesses were placed between the lead block and the source. With a 218 millicurie source 10 inches from the front of the ionization chamber it was just possible to detect (in the ionization current) the difference between 7 and 8 inches of steel. In other words, a hole 1 inch deep and ½ inch in diameter could have been detected in 8 inches of steel. Continuing, it was found that a difference could just be detected between 5 and 5½ inches of steel.

In any ionization method the width of the chamber slit is a limiting factor. To detect small imperfections a very narrow slit would be necessary, requiring very high sensitivity in the ionization apparatus. In addition, extremely good insulation would be required, a troublesome matter.

Since these experiments were made technique in the measurement of small currents has been developing rapidly. It is now possible to measure currents smaller than these with rugged and portable equipment. Any comparison of these results with the photographic results so far as sensitivity is concerned would accordingly be premature.

In order to obtain a record of the opacity of an object by any ionization method it would be necessary, however, to scan the area studied and to chart the variations in ionization current obtained. Such a method would be troublesome, though possibly some automatic scanning device might be worked out.

(b) As noted in the beginning of the paper thin sections of metal had been radiated by means of γ-rays and the emergent beam observed upon a fluorescent screen years ago by Rutherford. Repetition of this experiment showed the effect to be striking. A moment's consideration, however, is sufficient to discover the inadequacy in such a method, for only very thin sections may be successfully inspected and in such cases no radiographic method is needed unless it be the X-ray.

(c) Although at the beginning of this work it was thought that an ionization method of recording the emergent beam was to be preferred because of the speed with which a record might be obtained, it was found that the photographic method yielded surprisingly good results and the work continued by this method entirely. Agfa and Eastman double-emulsion X-ray films were used exclusively.

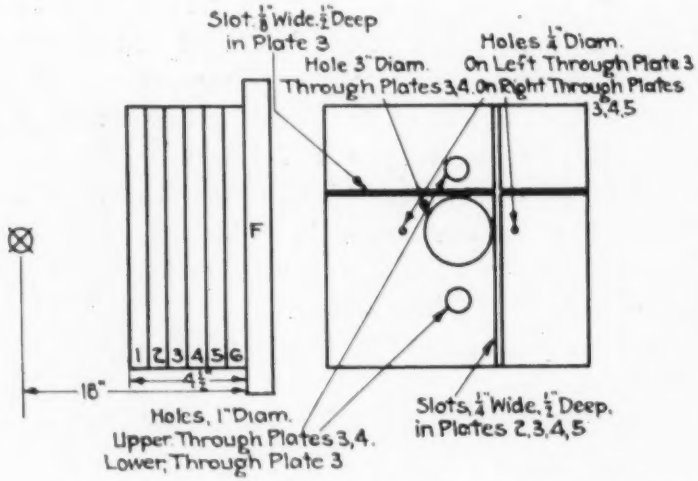


Fig. 16—Diagram Showing Method of Preparing Specimen Radiographed in Fig. 17.

Radiography by Means of γ-rays

In all several hundred photographs were taken of divers objects. The authors had at their command the radiographic technique so completely developed for X-rays, but little use was made of many of its details, for in a pioneer attempt, such as this, it is more economical of time to discover a few essential facts before much endeavor is made towards refinement. Of these many photographs only a selected few are reproduced here, chosen to demonstrate the general nature of the results obtained. The chronology of the experiments performed was not that indicated by the sequence of photographs in this paper, so that there is lacking the indication of improved quality, resulting from experience, observed during the course of the work.

Without any initial conception of exposure times it was inevitable that many photographs should show too little or too much exposure. This may be noted even in the photographs reproduced here, with a tendency toward over-exposure. For obvious reasons the shortest possible exposure time is desired. Some films were exposed "plain", that is, without the use of an enhancing screen, some using a Patterson calcium-tungstate screen, and some using a lead backing as an enhancing screen.

For these various reasons the exposure times noted for the



Fig. 17—A Series of Steel Plates, in Certain of Which Were Cut Holes of Several Diameters and Slots. Details of Arrangement Given in Fig. 16. (Film No. 8)

Distance, source to film:	18 inches
Intensifying screen:	Film between two Patterson screens
Time of exposure:	12 hours
Mean strength of emanation:	198 m.c. in one bulb

Results: Film much darker than necessary. Half the stated exposure time would easily have been sufficient. Contrast on Fig. 17 somewhat greater than on original film. Fogging shown on upper right-hand corner of Fig. 17 only very light on original film. Film covered only part of detailed plate in Fig. 16, as may be seen by a comparison of Figs. 16 and 17. Smallest hole in left is of particular interest. It appears very strongly on Fig. 17, though it is only ¼ inches in diameter and ¾ inches deep, showing that the γ-ray method in this case brought out strongly a defect ¼ inches in diameter and 17% of the total thickness deep. A similar sensitivity is shown by the lower 1-inch hole. The sensitivity of the method is shown also by the following film.

various photographs must be regarded carefully.[20] Considering also the fact that most of the films were over-exposed it is obvious that the exposure times noted could have been less.[21]

[20]Estimations of this reduction in exposure time vary. The booklet, "Der gegenwärtige Stand der Röntgendurchstrahlung von Metallen" published in 1928 by Siemens-Reiniger-Veifa, states (page 9) that the exposure time may be shortened to from ⅛ to 1/100 dependent upon the wave-length of the ray, the shorter rays giving a proportionately greater saving in time. This would indicate a great saving for γ-rays but as yet no determination has been made.

[21]It is obvious that any development in photography toward shortened exposure times would be of the greatest importance to γ-ray (or X-ray) radiography. Recent experiments in this Laboratory indicate the development of a method which will reduce the exposure time to at least one-half of that now necessary.

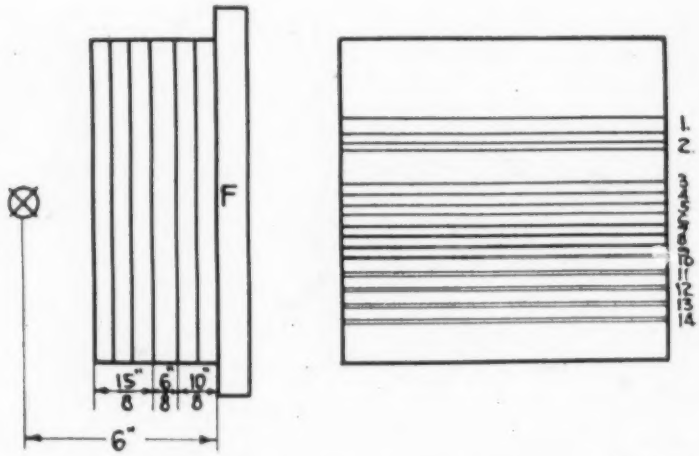


Fig. 18—Diagram Showing Method of Preparing Specimen Radiographed in Fig. 19. Dimensions of Slots Were as Follows:

Number	Width in Mm.	Depth in Mm.
1	13	13
2	6	13
3	1	2
4	1	3
5	1	6
6	1	12
7	2	2
8	2	3.5
9	2	6.5
10	2	12

The specimens radiographed were chosen to represent the important features commonly found in radiography. They include "synthetic" specimens, that is, specimens built up of plates with holes, slots and so on simulating cracks, voids, and inclusions actually found as defects in metallurgical materials. It was considered best to work first with such synthetic specimens, since the utter lack of previous work made all results uncertain. In addition to such specimens there are included radiographs of a cast iron block with defects, a steel casting with surface cracks, a steel casting with sand inclusions, a bronze casting, and two imperfect welds.

It should be clear, from what has been said, that the necessary equipment and technique is extraordinarily simple. Many of the photographs were taken upon the table illustrated in Fig. 6. The thistle tube rising from the center of the table held the radium emanation. Specimens were distributed radially around this as indicated by the guides on the table top which served to orient the specimens and the photographic film holders. The technique of taking γ-ray radiographs consisted simply in mounting the metal object to be photographed upon the stand (if the stand were used),

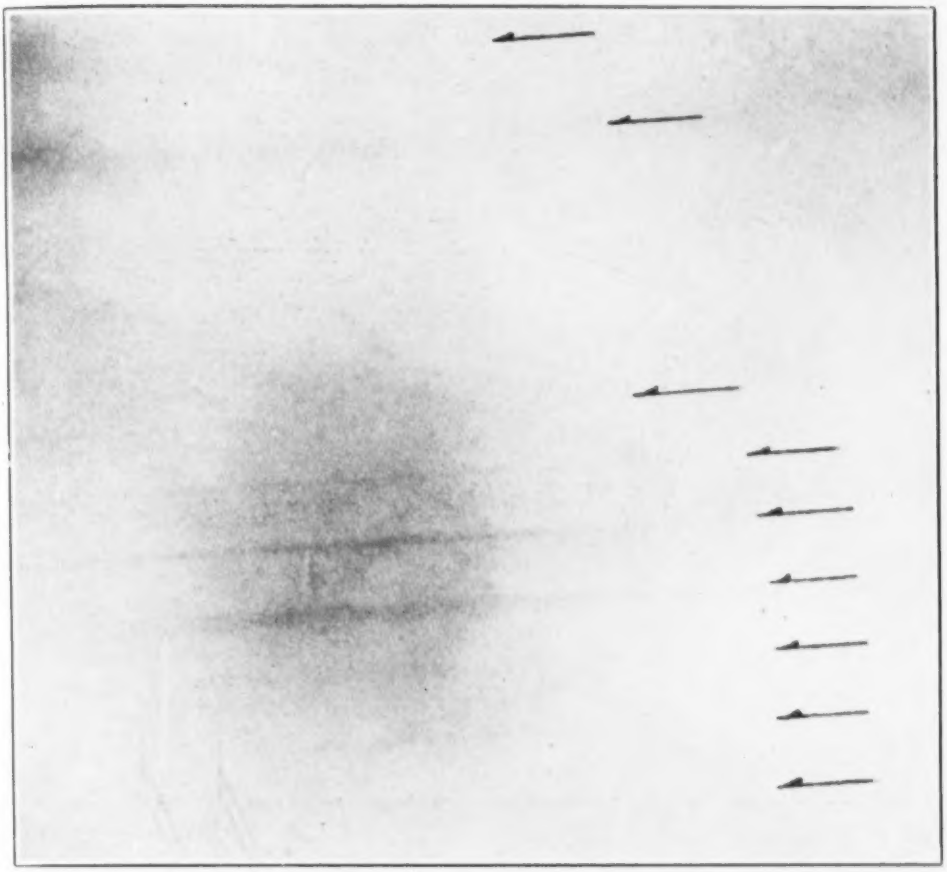


Fig. 19—A Series of Steel Plates, Total Thickness 3⅞ inches, in One of Which (marked "6/8" in Fig. 18) a Number of Slots of Varying Depth and Width Were Cut. Details Given in Fig. 18. The Slotted Side of the Plate was Turned Towards the Film. (Film No. 9)

Distance, source to film:	6 inches
Intensifying screen:	None
Time of exposure:	4 hours
Mean strength of emanation:	179 m.c. in one bulb

Results: This radiograph was taken to test the sensitivity of the γ-ray method for small defects. It must, of course, be borne in mind that the slots illustrated in Fig. 18 are well defined geometrically, which is favorable to the test. The original film was nearly correctly exposed, though the use of Patterson screens would have decreased the exposure time. Owing to the close proximity of the source, there is a greater density in the center of the film than at the edges. The great divergence of the rays at the edges likewise had the effect of spreading out the registration of the slots, and this effect, coupled with the diminution of intensity of the ray, made certain of the slots far from the center not visible on the film.

Especial attention should, however, be given the five slots registered near the center of Fig. 19. The third arrow from the top indicates the photographic result from slot No. 3 in Fig. 18, which was 1 mm. wide by 2 mm. deep. Under the conditions of this experiment, therefore, a defect 1 mm. wide and only 2 per cent of the total thickness deep was detected.

The second arrow from the top indicates the photographic result of slot No. 2. Slot No. 1 is only faintly indicated (top arrow) for the reasons stated above.

aligning it with the thistle tube, adjusting the holder containing the film at the rear of the object, dropping the metal capsule containing the bulb of radium emanation in the thistle tube, and receiv-

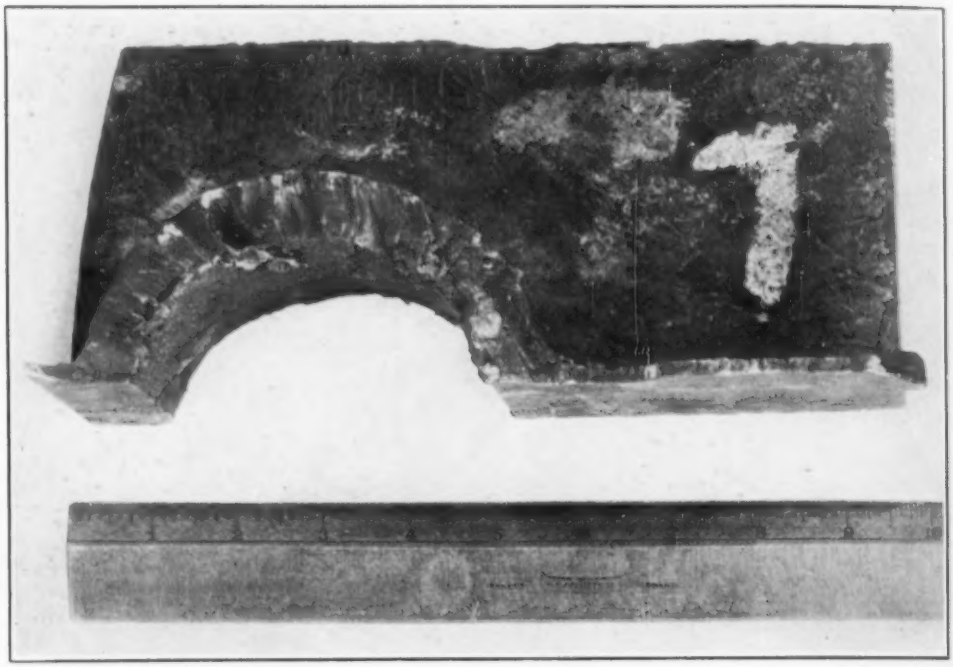


Fig. 20—Steel Casting 1-Inch Thick with Cracks. Radiographed in Figs. 21 and 22.



Fig. 21—A Steel Casting, 1 inch Thick, with Cracks, as shown in Fig. 20. This is an X-ray Radiograph. Fig. 21 is not a Very Satisfactory Reproduction of the Original Film. Contrast Printing Has Given Fig. 21 a Nonuniform Appearance. The Edges of the Piece are Less Distinct Than on the Original Film. The Number "3" is Merely an Identification Mark. Contrast Printing has Increased the Contrast in the Cracks. (Film No. 10)



Fig. 22—Same as with Film No. 10 (Fig. 20).

Distance, source to film:	66½ inches
Intensifying screen:	None
Time of exposure:	10½ hours
Mean strength of emanation:	2181 m.c. in six bulbs

Results: Film exposed more than necessary. Exposure at a shorter distance and by the use of Patterson screens would have made a shorter exposure time and a weaker source of radiation sufficient.

Film No. 11 is, of course, to be compared with Film No. 10 (It will be noted that Film No. 10 should be reversed, so that "3" reads correctly in order to match it with Film No. 11). It may be seen immediately that the cracks showing on Fig. 10 are much darker than those on Fig. 11. This is the familiar diminished contrast mentioned previously. It will also be noted that certain cracks (above the arch) visible on Film No. 10 cannot be found upon Film No. 11. It is uncertain whether the apparently lower sensitivity towards cracks is inherent in the γ-ray method (owing to the lower absorption coefficient) or whether it resulted in this case from an inferior alignment of the direction of the crack with respect to the beam in the case of the γ-ray radiograph.

ing the film holder at the expiration of the exposure time. The films used and the developing procedure were similar to those in X-ray radiography.

It is an important feature of this method that many specimens may be arranged around the γ-ray source, for the radium emanation emits γ-rays in all directions, that is, spherically. In appraising the cost of such a method of inspection this feature must be remembered.

γ-Ray Radiographs

The descriptive matter accompanying the radiographs needs a word in explanation.

The technique described in the third paragraph above was always

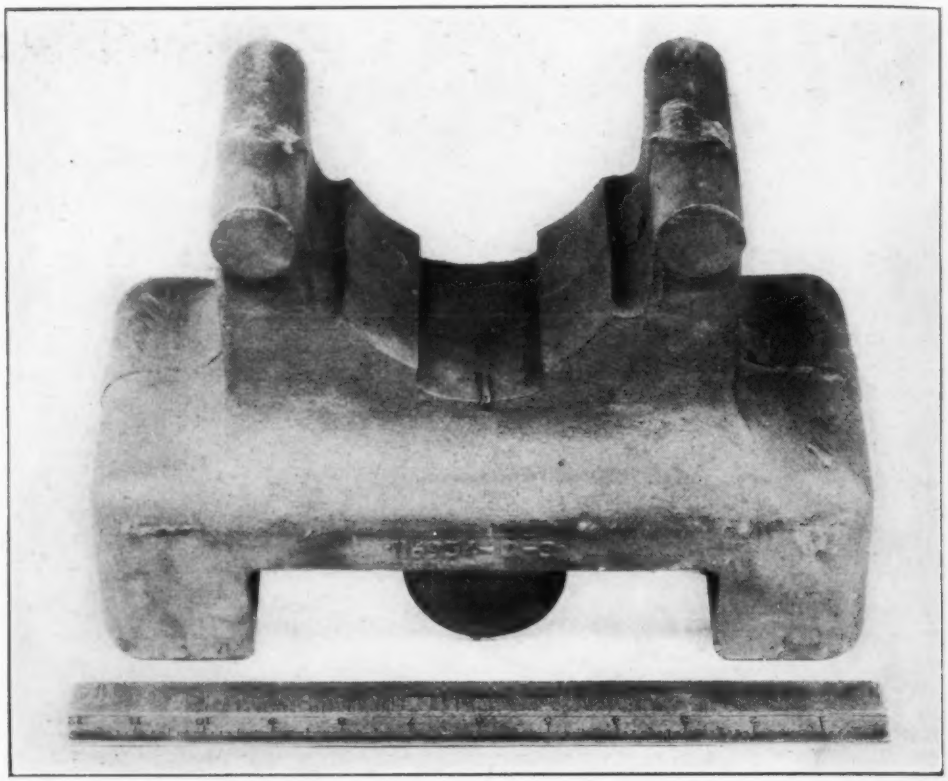


Fig. 23—Bronze Casting Radiographed in Fig. 24.

used, except that certain objects were photographed upon the floor. Diagrams of the relative positions of the emanation, object, and film holder are given only when necessary.

"Distance, source to film", refers to the distance, in inches, from the bulb of radium emanation to the film holder. The information listed after "intensifying screens" is self-explanatory, as is that after "time of exposure". "Mean strength of emanation" refers to the radiating strength of the emanation half-way through the exposure. The strength of the emanation was therefore somewhat greater at the beginning of the exposure and somewhat less at the end than that of the value given (see Fig. 1). Under "Results" are listed a few essential features in the radiograph to which it is desired to call attention. Several of the γ-ray radiographs are accompanied by X-ray radiographs of the same object for purposes of comparison.

The radium emanation was held in a small ($\frac{1}{8}$ inch in diameter) glass capsule, enclosed in a brass sheath, $\frac{1}{2}$ inch in length, $\frac{3}{16}$ inch in diameter. In several cases, when strong sources were used, more

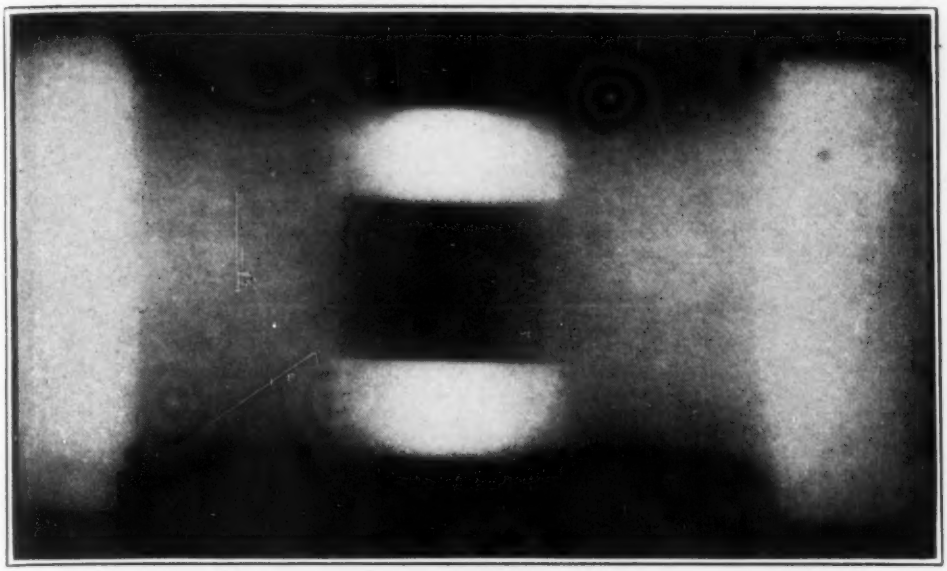


Fig. 24—An Irregular Bronze Casting, Pictured in Fig. 23. (Film No. 12)

Distance, source to film:	26 inches
Intensifying screen:	None
Time of exposure:	10¾ hours
Mean strength of emanation:	2100 m.c. in six bulbs

Results: Film exposed somewhat more than necessary. Exposure time and strength of emanation could have been further reduced by use of Patterson screens. Absorption of bronze is greater than that of steel requiring a somewhat stronger source or a somewhat longer exposure time. A picture taken simultaneously, with film between two sheets of lead foil gave slightly better contrast but approximately the same density. Note small flaw in upper right corner of casting.

than one of these containers were used, as will be noted. The use of several containers is not to be recommended in general, since it increases the dimensions of the radiating source and therefore operates against a high degree of definition in the resulting radiograph.

The usual difficulty in photographic reproduction of the original radiographs for publication was experienced, perhaps somewhat more severely, for the γ-ray radiographs do not show so great a contrast as the ordinary X-ray radiographs. The films were mounted on wooden frames, inserted in a light-tight viewing cabinet, and photographed with a brilliant source of illumination at the rear. Best results were obtained with process plates (Wratten M) and with printing upon contrast paper (Azo glossy, usually No. 4). It is inevitable that the published reproductions of the original radiographs should be unsatisfactory. In each case under "Results" is given a few sentences comparing the reproduction with the original film.

The first three films, respectively of an echelon of steel plates,

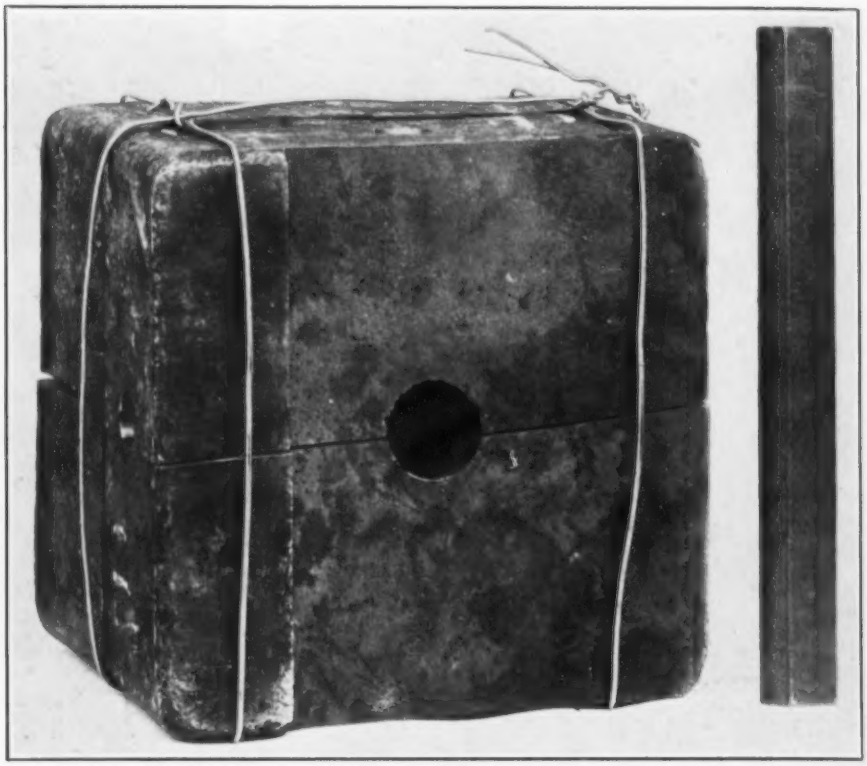


Fig. 25—Block of Cast Iron With a Large Hole Leading Off One Face. Assembled After Cutting and Radiographed in Fig. 27.

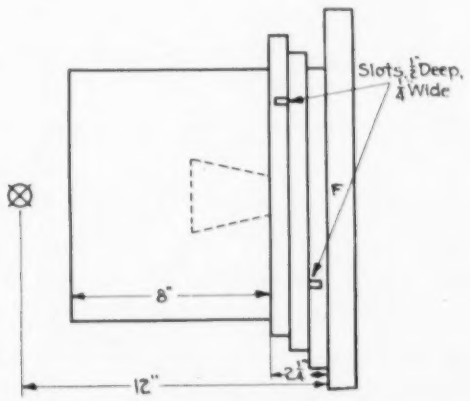


Fig. 26—Diagram of Method of Preparing Specimen Shown in Fig. 25 and Radiographed in Fig. 27.

a lathe tool rest, and a wrench, were taken merely to test the sensitivity of the γ-ray radiographic method for detail. These are all within the X-ray range of thickness.

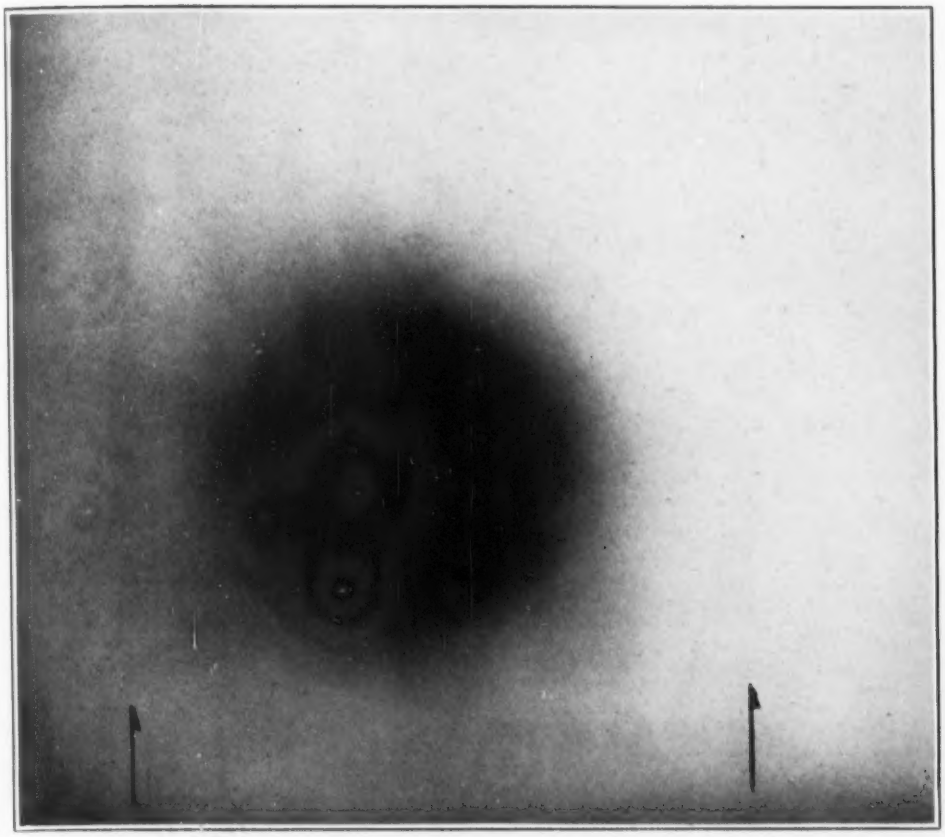


Fig. 27—A Block of Cast Iron with a Large Hole Leading Off One Face (Fig. 25, Assembled After Cutting), with Three ¾-inch Steel Plates with Slots, as Shown in Fig. 26.

Distance, source to film:	12 inches
Intensifying screen:	None
Time of exposure:	10¾ hours
Mean strength of emanation:	2100 m.c. in six bulbs

Results: Film nearly correctly exposed. Photographic reproduction of Film No. 13 made by photographing film laid upon sheet of white paper. This was found to be a very satisfactory way of viewing negatives that are not too dense.

Large hole very clearly visible. Also slots in plates may be seen to right and left of large hole, indicated by arrows in Film No. 13. Exposure time and strength of emanation could have been reduced considerably by use of Patterson screens. One inch hole in top and another in bottom were drilled after taking radiographs to facilitate cutting up the specimen so naturally do not appear in any radiographs.

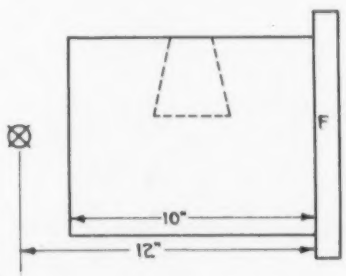


Fig. 28—Diagram of Method of Preparing Specimen Radiographed in Fig. 29.

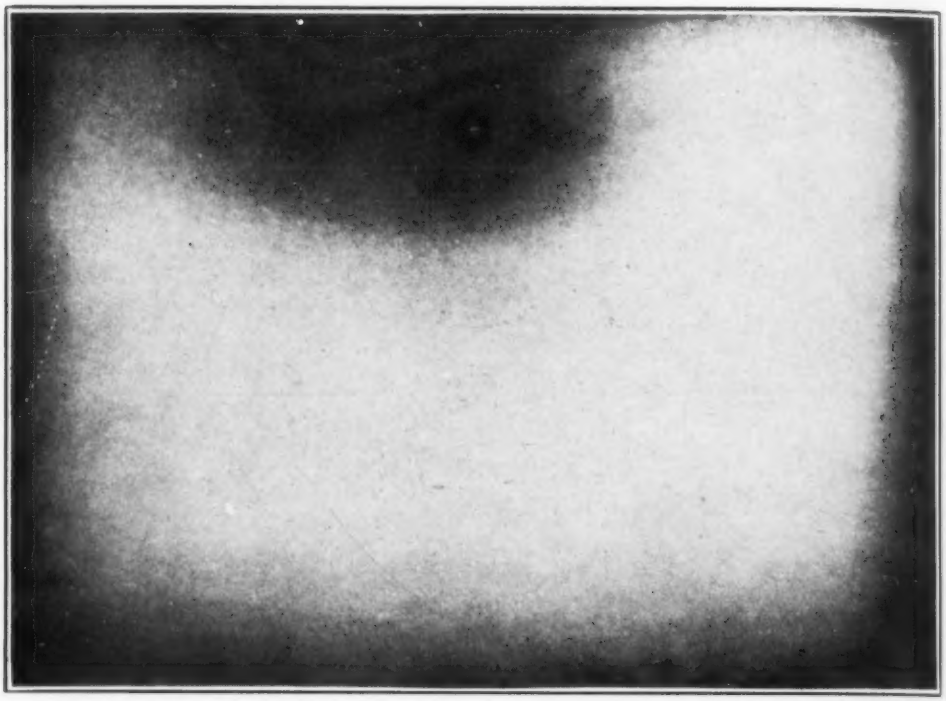


Fig. 29—Same as with Film No. 13 (Fig. 27), Except Without Steel Plates. Hole on Side of Casting as Shown in Fig. 28.

Distance, source to film:	12 inches
Intensifying screen:	One Patterson screen
Time of exposure:	8 hours
Mean strength of emanation:	1150 m.c. in two bulbs

Results: Original film imperfect, giving a "spotty" radiograph. Hole in side clearly visible, but distorted owing to divergence of beam. Film badly overexposed.

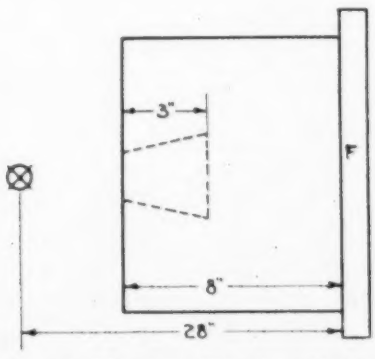


Fig. 30—Diagram Showing Method of Preparing Specimen Radiographed in Fig. 31.

Discussion and Summary

The aim of the present work may be stated in simple terms: it is an attempt to appraise in a preliminary and in a purely scien-

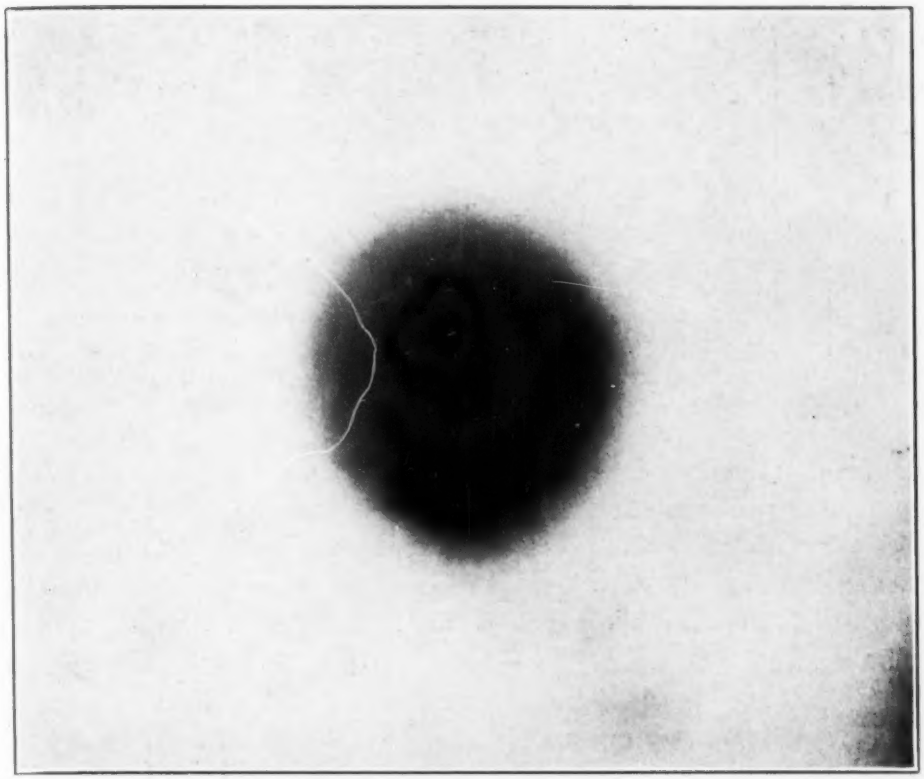


Fig. 31—Same as with Film No. 14 (Fig. 29). Hole Towards Source, as Shown in Fig. 30. (Film No. 15)

Distance, source to film:	28 inches
Intensifying screen:	One sheet of lead foil
Time of exposure:	10½ hours
Mean strength of emanation:	2181 m.c. in six bulbs

Results: Exposure greater than necessary. Larger hole may be clearly seen. In addition to this there may be discovered in Film No. 15 a cloud-like image floating over the large hole and downwards slightly to the right. This suggested a defect in the back of the block. Upon cutting the block the defect was found and may be seen as small holes in the small pieces in Fig. 32.

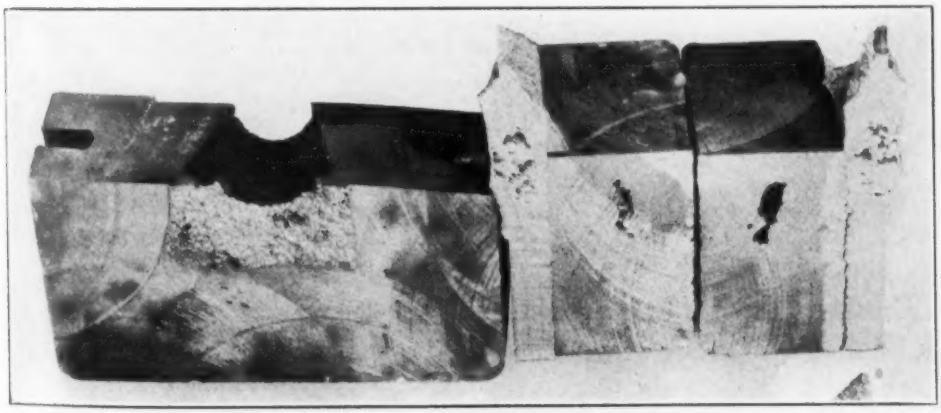


Fig. 32—Showing Defects in Block.

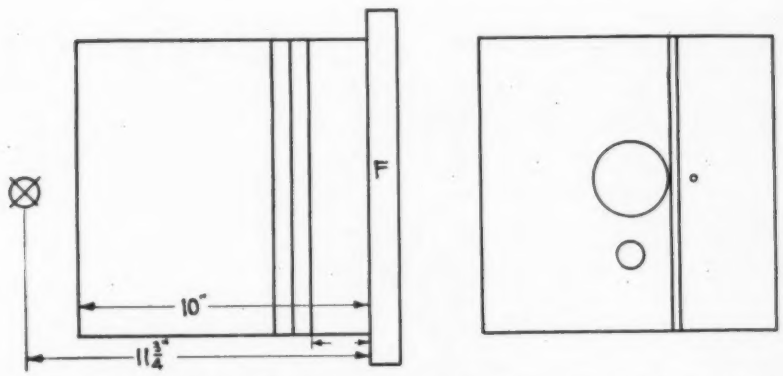


Fig. 33—Diagram Showing Method of Preparing Specimen Radiographed in Fig. 34. (Note: The Missing Dimension is 1⅛ inch).

tific fashion the use of γ-rays for the radiographic detection of defects in metallurgical material. It does not, therefore, present a finished testing method to industry; nor is there any attempt to analyze the economics in any possible commercial exploitation. This latter task must be left to those whose interest might make such a testing method desirable.

The results attained are those illustrated by the γ-ray radiographs and itemized under "Results" in the appended descriptions. To complete the appraisal possible at this time these results should now be recapitulated; and furthermore the practical features surrounding the use of a γ-ray method of inspection should be set forth.

The physics of γ-rays (developed from the standpoint of radiography) in the introduction indicates that γ-rays, because of their shorter wave-length (Fig. 2) and correspondingly lower absorption coefficient, should be able to penetrate thicknesses of metal with a smaller loss of intensity than X-rays, and, moreover, for the same reason, should not require for great thicknesses the relatively tremendous increase in exposure time characteristic of the X-ray wave-lengths now commonly in use.

It is a corollary that the transmitted beam should register defects upon a photographic film with a contrast inferior to that obtained with the present radiographic X-ray wave-lengths. This diminished contrast is an inevitable resultant of the shorter wave-length, and is, of course, not peculiar to the radiation originating from the decomposition processes in radium emanation; development of higher voltage X-ray radiography would lead to a similar circumstance.

It was also indicated in the introduction that the scattering of

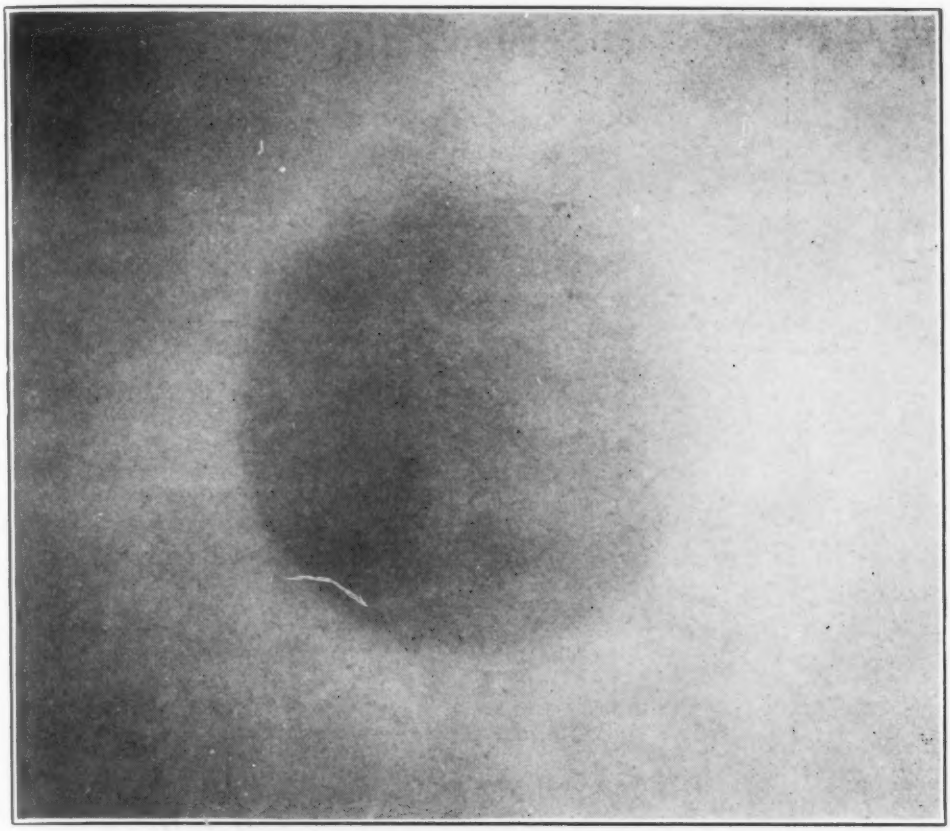


Fig. 34—Pile of Steel Plates, Total Thickness 10 Inches with Holes and Slots in Two Plates as Indicated in Fig. 33. The Positions of the Two Plates Bearing the Holes and Slots Are Indicated in the 10-Inch Section in Fig. 33; the Rest of the Section was Plain Plates. (Film No. 16)

Distance, source to film:	12¼ inches
Intensifying screen:	None
Time of exposure:	10¾ hours
Mean strength of emanation:	2100 m.c. in six bulbs

Results: Underexposed. Another film taken simultaneously with two Patterson screens, had nearly correct density for viewing by reflected light when backed by white paper; by mistake the poorer of the two was sent for reproduction. On both, the hole 3 inches diameter and 1½ inches deep are shown clearly and the 1-inch hole 1½ inches deep barely seen; on the better film both slots are faintly recorded. The ¼-inch holes do not appear. A single bulb with a correspondingly longer time would have considerably improved the definition in this and several other films.

γ-rays (Fig. 4) should not be inimical, and that, in fact, the almost complete absence of "back-scattering" (scattering at an angle of 180 degrees and thereabouts to the primary beam) is an especially favorable feature of γ-rays, for it is unnecessary to take the precautions (various types of shielding) which are a necessary part of X-ray radiography. It may be said that the experiments performed appear to bear out these views.

Radiographs have been taken of objects the thickness of which exceed that thickness now possible to radiograph with X-rays within

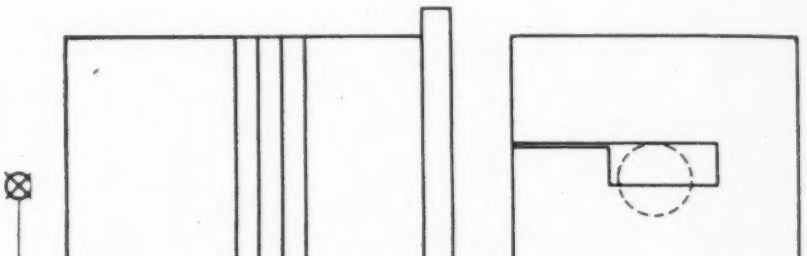


Fig. 35—Diagram Showing Method of Preparing Specimen Radiographed in Fig. 36.

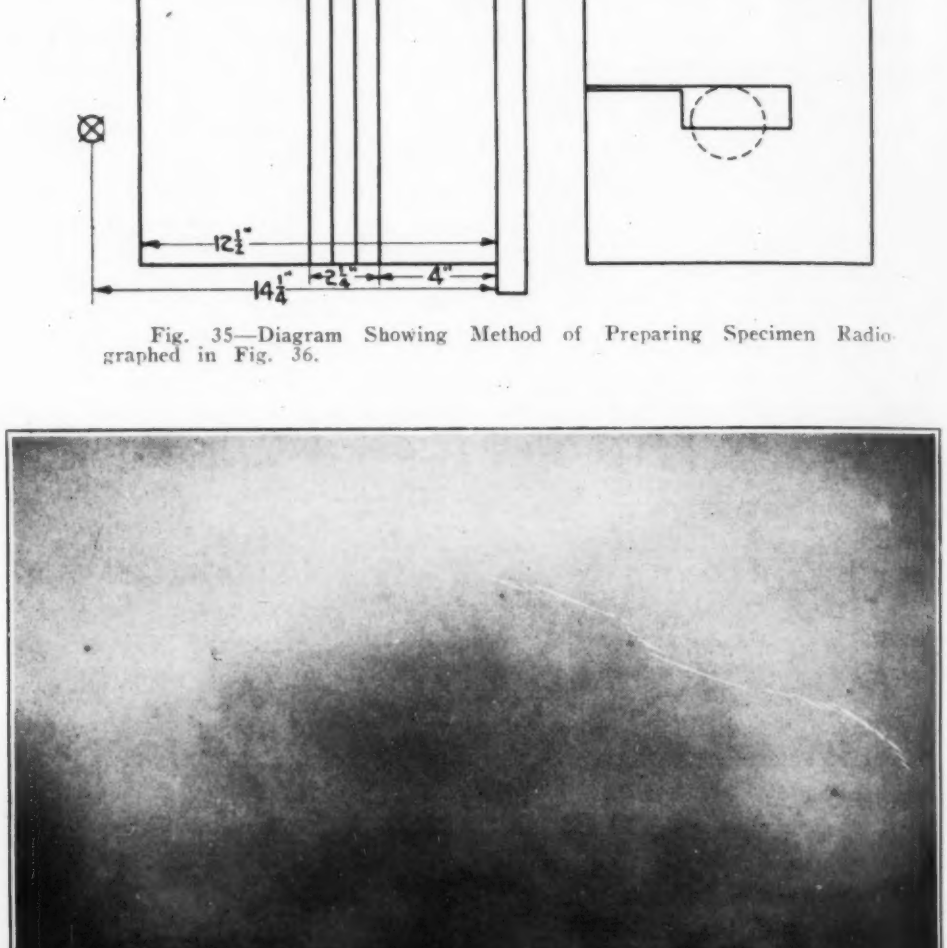


Fig. 36—Series of Steel Plates, Total Thickness 12½ Inches, All Plates Plain Except Three, Near Center, as Indicated in Fig. 35. Two of These Were the Plates Illustrated in Fig. 33, the Third Illustrated on the Right of Fig. 35 Which Shows the Alignment. (Film No. 17)

Distance, source to film:	14¼ inches
Intensifying screen:	One Patterson screen on each of two films
Time of exposure:	10½ hours
Mean strength of emanation:	2181 m.c. in six bulbs

Results: Underexposed. The photographic reproduction was done with rear illumination with two films (taken simultaneously) superimposed. The films were held at an angle during reproduction in an attempt to bring out the dark area in the center more strongly. Within the range of intensities and exposure times available during these experiments, this film shows that the thickness for satisfactory radiographs has been exceeded. Our opinion is, however, that a longer exposure and a source of smaller dimensions would allow this and probably greater thicknesses to be radiographed.

reasonable exposure times, that is, from four to ten inches of steel. The minimum exposure times for such thicknesses have not been

defined. If it should appear desirable to develop a γ-ray radiographic method for practical use it will be necessary to work out a correlation of strength of source, time of exposure, and photographic result. In such an eventuality especial attention will have to be given to methods by which the exposure time, at best rather long with γ-rays, can be reduced to a minimum.

As to be expected, enhancing screens (of calcium tungstate) are very effective in reducing exposure times, though the actual saving in time has not been defined. Lead foil likewise proved effective in shortening exposure times. The use of multiple films is effective in making visible to the eye dimly developed images, and this technique is to be recommended to eliminate from consideration possible spurious effects originating in defective films. It has been found very useful to view the developed film in good light upon a piece of white paper. The enhancing effect of this device is startling. Film No. 13 (Fig. 27) was photographed in this way.

The diminished contrast is illustrated especially by Figs. 14 and 15 and Figs. 21 and 22. This diminished contrast is not necessarily an argument against the use of γ-rays, especially in the upper ranges of thickness, but it means that importance must be given to slight density discontinuities on the developed film. No detail shown in Fig. 14 is absent from Fig. 15; it merely appears with diminished contrast. This diminished contrast orginating in a lower absorption coefficient has one advantage: it facilitates the radiography of irregular sections, for it is an easier matter with γ-rays to obtain the whole of an irregular object at a correct exposure than it is with X-rays. This, likewise, is shown by Figs. 14 and 15.

Although no efforts were made to prevent scattering in these experiments, no poor results were obtained which could be ascribed to this cause. Thus Films No. 3, 11, 15, and 17 were made simultaneously as were Films No. 12, 13, and 16 with the objects closely together and several of them of very considerable mass, yet fogging appears to be unimportant on these films. This, taken with the fact that portions of the film exposed to the primary beam are not so badly overexposed when γ-rays are used as when X-rays are used, explains the lack of halo at the edges of the specimens.

The sensitivity of the γ-rays towards small defects seems satisfactory, though in the absence of extensive experimentation there may be some uncertainty on this score. Despite the fact that Film No. 9 was taken upon a "synthetic" specimen, the certain identifica-

tion of a slot only 2 per cent of the total thickness is promising. The discovery of the defect in the cast iron block upon Film No. 15 was, of course, somewhat gratifying.

The above discussion is all built upon the photographic registration of the transmitted beam. For reasons already given it is believed that fluoroscopic methods are not feasible and that at present the photographic method is superior to methods by which the intensity of the transmitted beam is registered by means of its ionizing effect.

The practical features surrounding the use of γ-rays for radiography are so greatly different from those of X-rays that care must be taken in comparing practical application of the two methods.

The technique of γ-ray radiography is extremely simple. There is required merely a source of γ-rays of suitable strength (radium or radium emanation) mounted simply (Fig. 6, for example), the object to be radiographed placed at a suitable distance,—defined by the strength of the source, the thickness of the object, the exposure time desired, and the definition required—and a photographic film, with or without intensifying screens, placed behind the object.

During the exposure no attention need be given and the operator may leave, returning at the expiration of the necessary exposure time. There is, of course, no high voltage equipment to be attended, nor are there any permanent high voltage leads to the source. In fact, the set-up could be arranged at almost any conceivable place, for everything is entirely portable and rugged.

The emission of γ-rays cannot be controlled. Whether or not the radioactive source is being used for radiographic purposes, it will continue to emit γ-rays. It would be advisable, therefore, to make as continuous use of the source as possible during the time it is in the possession of the operator.

Since γ-rays are emitted in all directions—that is, spherically—the source might be completely surrounded by objects to be radiographed. Because of the inverse-square law governing the diminution of the intensity with distance from the source, it is unimportant whether a large number of objects be radiographed at a considerable distance with a long time of exposure, or a small number at a short distance with a brief time of exposure; in either case the profitable use made of the γ-ray would be the same, with the qualification that a greater degree of definition would be obtained at the greater distances.

With a constant source of γ-rays and a large number of objects of varying dimension to be radiographed suitable exposures might be obtained either by varying the time of exposure, or by placing the thinner objects at greater distances and keeping a constant time of exposure for the whole arrangement. This latter scheme was followed in preparing Films No. 3, 10, 15, 17 and Films No. 12, 13, 16.

In the use of γ-rays, as in the use of X-rays, protection must be given the operator against the harmful effects of the rays. It would hardly be useful here to describe in detail the precautions the medical profession has found necessary, except possibly to mention those affecting the technique of operation. Distance, of course, is the best protection, and since no attention during exposure is required, this requirement may easily be met. The emanation is usually transported in a small but heavy-walled lead box and the emanation handled with forceps. These are probably the only details to be mentioned. The authors have been assured by medical practitioners that there should be no objection to the use of γ-rays for the purpose suggested here originating in possible harmful effects to the operator.

Acknowledgment

The authors wish to express their obligation to a number of persons who rendered valuable assistance during the course of this work: to Dr. Howard A. Kelly, of the Howard A. Kelly Hospital, Inc., of Baltimore, Md., who granted us permission to make use of his supply of radium emanation, and especially to Dr. F. West, superintendent of the hospital, who attended to our various needs during the work with the greatest of consideration and helpfulness; to Prof. A. F. Kovarik, of the Department of Physics, Yale University, who discussed the problem with us previous to the beginning of the work; to Dr. H. H. Lester, of the Watertown Arsenal, Watertown, Mass., who rendered assistance from time to time during the work; to Prof. J. B. Hoag, of the Department of Physics, University of Chicago, who made some preliminary experiments for us upon photography with γ-rays; to J. T. Gow, of this Laboratory, who lent assistance in many ways during the prosecution of the work; and finally, to Captain E. G. Oberlin, U. S. N., whose sympathy with research made this work possible.

DISCUSSION

Written Discussion: By William P. Jesse, research laboratory, General Electric Co., Schenectady, N. Y.

The authors are to be congratulated on these experiments in the use of γ-rays for radiography. Considering the inherent difficulties as regards contrast, due to the hardness of the rays and the usual difficulty that printed reproductions are never as good as the negatives themselves, it is astonishing to see the amount of detail shown in some of the plates.

I think that the authors, being interested in the scientific aspects of the problem, have quite justly ignored economic considerations in their discussion of their results. However, such economic considerations must enter in when one considers the applicability of the method to commercial testing.

Judging from the millicuries recorded in some of the experiments, it would seem that the writers had at their disposal the emanation resulting from some two to three grams of radium. Such a stock of radium would be prohibitive in price to the average company desirous of using γ-rays in this way. Leaving out the possibility of coöperative use of a radium supply, it would appear that the next advance would have to be the increasing of the efficiency of the operation by the use of improved films, intensifying screens, etc. The authors seem to hold out hope of improvements along these lines, and so I should like to know, in the light of their latest knowledge, how much, in general, they feel the exposures recorded might be reduced by such improved technique.

Written Discussion: By Peter P. Alexander, Thomson Research Laboratory, General Electric Company, Lynn, Mass.

A study of this outstanding contribution has indicated that it was written in such a scholarly manner and supplied with such complete data that a discussion is perhaps superfluous.

It is always possible, of course, to find one or two points on which one may have differing opinions. One may argue that with improved technique it is possible to collect daily from one gram of radium a volume of emanation possessing a strength of 166 millicuries instead of 160, as reported; or that the mean strength of emanation was excessive in taking certain radiographs. But such arguments will hardly be warranted at this time, nor will they add anything to the clarification of the subject presented by the authors.

Perhaps it will be more profitable to say a few words about the method of preparation of the emanation which is the source of the γ-rays.

As the authors acknowledge, the fundamental facts underlying this method of radiography have been known for years. Professors Rutherford and Soddy, in their original investigations, have used all three reported methods of detection of the γ-rays. But the results obtained were purely scientific facts which remained in the highly specialized laboratory beyond the reach of engineers and practical metallurgists. Up to this date the only known attempt to use γ-rays for the detection of flaws in castings was reported about two years ago from Leningrad. Even that was apparently limited to electric methods of detection. The present paper, therefore, comes really as the first concrete data on the detection of flaws in metallurgical materials by the use of radium emanation and the photographic plate.

Whatever may be the immediate future of this method of testing, we will all agree that today we have heard the description of a new method of testing which can be developed into a tool of tremendous importance.

To appreciate more fully the splendid results obtained by the authors, we must visualize the delicacy of the technique involved in the use of radium emanation and the active nature of this wonderful substance. The maximum volume of that gas which may be obtained at one time from a gram of radium is 0.6 cubic millimeter, and if the pumping is done daily, as is customary, the amount of gas which the operator may finally seal into a glass bulb is a small fraction of the volume of a pinhead. The process of "pumping" in itself is a series of delicate laboratory operations.

If the radium bromide, which is generally used instead of pure radium, is kept dissolved in water, it decomposes the water into oxygen and hydrogen. The result is that the infinitesimal volume of evolved radium emanation is mixed with much larger volumes of hydrogen, oxygen and a certain amount of carbon dioxide. This dilution of the emanation is very helpful, as it facilitates pumping. Yet, in further operations, the emanation must be separated from these gases. This involves the use of the mercury Töpler pump, induction coil and a supply of liquid air. Finally, when all these difficulties are overcome and the pinhead of emanation is prepared, it rapidly decays, with the result that in three weeks it is worthless for any experimental work with γ-rays.

One may question, then, why instead of going to all this trouble of preparing the emanation, radium salt itself is not used. There are several reasons for this. One is the tremendous cost of radium, which, at the present time, is in the neighborhood of $100,000 per gram. The emanation, however, is comparatively cheap, since it is evolved by radium continuously, regardless of whether it is used or not, and also without reducing the radioactivity of the radium in any appreciable way.

Another reason is that the emanation is much more radioactive than radium itself. A gram of radium continually evolves energy equal to 33 calories per hour, and the fraction of one cubic millimeter of emanation which forms, so to speak, a thin atmosphere around it, evolves 100 calories per hour. Mass for mass, therefore, the emanation is an incomparably more radioactive substance than radium.

As Professor Soddy expressed it, one pint of emanation is equivalent to one-half ton of pure radium. This gives an idea of the active nature of the gas used in the above experiments.

It is true that the emanation has been supplied to the authors by Dr. H. A. Kelly, eliminating from their task this initial work of preparation, yet if this method is used for the regular testing of metallurgical materials, a special laboratory supplied with at least one gram of radium and all the equipment for preparing the emanation should be provided. However, even if the emanation is supplied already prepared, the further handling of the tiny capsules containing this wonderful substance should be done with great care, especially if the experiments necessitate doses having the tremendous strength of 2100 millicuries. To start with, no finger of the operator should

ever touch the capsule for fear of contracting burns which are not felt and do not appear until several days later. Happily, the normal tissues are very much more resistant to the radiations than the pathological ones. As has been shown by Janeway and Weing, to destroy the normal epithelial cells requires a dose four times stronger than that sufficient to destroy the carcinomatous cells. This selective property, by the way, is the secret of success in the use of radium emanation in the treatment of deep-seated cancer. Nevertheless, the operators must be constantly on guard against the radiations which, unlike X-rays, are issued continually in all directions.

The results obtained by the authors are self-explanatory because of the remarkable radiographs published in their paper. One may see how with an adequate technique the radiographs giving very good contrasts can be obtained.

The great penetrability of the γ-rays which may cause trouble in obtaining very sharp contrasts can be utilized to very great advantage in the examination of castings of irregular shape.

It is possible to imagine that in the future by placing a small capsule of emanation inside the steel casting, such as a high pressure hydraulic valve, which is painted on the outside with suitable paint sensitive to γ-rays, the radiograph of the whole structure could be obtained with one exposure taken over night.

It is too early, however, to speculate on the practical results which will undoubtedly follow in due time the pioneer work described in this paper.

One may only hope that the authors will continue their investigations and extend them also to other modifications of radiography. For example, especially valuable results may be expected in the application of γ-rays to the monochromatic-pinhole method of investigation. If the difficulties of the suitable screening and directing of the γ-rays into separate beams can be overcome, it will be possible to obtain diffraction patterns through thicknesses very much greater than is possible at present. This will give us a valuable method of study of the strains and grain structure of metal in the vital parts of engineering structures without destroying them.

In conclusion, it may be stated that this outstanding contribution merits careful study by every engineer dealing with the testing of materials, and that it will be an inspiration to many research workers.

Written Discussion: By L. W. Spring, chief chemist and metallurgist, Crane Co., Chicago.

While the use of γ-rays for radiography of steel manifestly is in the experimental stage, this application is of great interest and of considerable promise. Some manufactured products of steels, such as forgings, castings, welded parts, etc., have been studied and some even inspected by X-ray. This latter method does very well where steel sections are not too thick and on articles of such shape that the X-ray tube and the film holding cassettes can be reasonably well placed. The greatest difficulty with X-ray is that commercially, sections not more than 3 or 4 inches thick can be radiographed without excessive time of exposure and loss of detail.

Radiography by use of γ-rays appears to have advantage in that the

source can be carried into the foundry or shop, that it can be inserted into hollow castings or other articles, and by proper arrangement of X-ray film about the outside, radiographs could be taken nearly 360 degrees around an even wall section. The apparent greater depth of penetration with possibility of radiographing sections 8 to 12 inches thick, is a distinct advantage. With the high pressures and temperatures now planned for modern steam, oil refining, and some chemical plants, pipe, valves, fittings, etc., likely will be required with sections considerably thicker than 3 or 4 inches. The exposure times given in this paper, however, are altogether too long for commercial application in steel and equipment manufacturing plants, and much work apparently remains to be done to make the process thoroughly adaptable.

Written Discussion: By Wheeler P. Davey, professor of physical chemistry, Pennsylvania State College, State College, Pa.

In January, 1915, using a Coolidge tube, the writer published a radiograph of a casting about an inch thick. Later in the same year he radiographed, with difficulty a steel slab two inches thick. It is interesting to see "how the baby has grown," so that now, by the use of gamma rays, an 8-inch slab can be radiographed with comparative ease. It is a pleasure to congratulate the authors of this paper on such an excellent piece of work. Instead of the 100,000 volt X-ray tubes of sixteen years ago, the authors have, by the use of radium emanation, obtained results which would have otherwise required a one-and-a-half million volt tube. The advantages and disadvantages of gamma rays have been discussed in a scholarly manner. In order to have obtained such good photoengravings, the original radiographs must have been exceptionally good. I agree with the authors that, at least at present, the photographic method is to be preferred to the ionization chamber method.

E. W. PAGE:[1] I have listened very attentively to what Mr. Mehl has said regarding the use of radium and I hope what I have to say will not be considered as judicial. Most of my experience has been with the use of X-rays although we have deduced much information from the use of shorter wave lengths such as is produced by radium.

Time of exposure is not particularly vital and is dependent primarily on the quality of information that is desired. It may require an exposure of at least ten minutes for a thin sheet of aluminum and yet it is possible to penetrate 2½ inches of steel in the same length of time.

I am interested in this paper primarily from the effect of absorption and in seeing some radiographs with an endeavor to show small differentiations on the film. One in particular where there were great differences in thickness of material and showing a fairly equal amount of density on the film. One of the remarks Mr. Mehl made, if I heard correctly in the back of the room, was that it was possible to show in the thin sections small marks due to etching, and still show a density on the film through heavy sections. I cannot quite understand how that small difference in density can be shown on the same film when it is the difference in rates of absorption that produce the different densities on a film from which the information is obtained.

[1]General Electric X-Ray Corporation, Chicago.

It is true that there is much to be considered due to the effect of scattering. We are able to eliminate the objectional effect of scattering and, as a matter of fact, we utilize much of the scattering effect to produce information on a film, so it is not so objectionable as one might think.

It might be interesting to know that X-rays are being produced with approximately a million volts in the laboratory at Schenectady. X-rays produced at this voltage have wave lengths in the same range as the gamma rays but with an energy equivalent in the neighborhood of ½ ton of radium. We intend to do some work, from a radiographic standpoint, with this equipment. How it will lend itself in producing radiographic information, I am not at this time able to say.

R. S. ARCHER:[2] I merely want to ask the authors a few questions which they may choose to reply to in writing. We have been employing radiography for several years on a strictly commercial basis. As far as we know, it is the first commercial installation in a metal fabricating plant, and its use has been quite successful. There is little question that in the case of sections which are too thick to penetrate with X-rays now available, the use of γ-rays is the only method that can be used. The authors have certainly done a great service by bringing that possibility to our attention. I am particularly interested in the comparison between the use of radium and X-rays within the limits of application of present X-rays, which is up to about 220,000 volts.

I would like to ask a few specific questions. (1) as to the quantity and cost of radium which would be equivalent to a 220,000-volt tube, or to a 90,000-volt tube, with special reference to materials and section thicknesses which can be properly studied by means of those tubes; (2) as to the technique of preparation of the radium emanation and putting this emanation in capsules ready for use; (3) as to the possible danger from the use of emanation and means of protecting operators and other people who may be exposed to this radiation.

The authors have stated that contrast is somewhat less with γ-rays than with X-rays. They have at the same time stated that the sensitivity of the two methods is about the same. This seems a little inconsistent inasmuch as there are certainly features of structure which are shown on X-ray radiographs in which the contrast is just barely sufficient to make the features visible. Because of the greater penetration of the γ-rays and less scattering, there might be some reason to think that the photographs would be sharper even though they might lack contrast. More information on this would be of interest. Finally, as to the photography of irregular objects, Dr. Mehl has pointed out what I understand to be an advantage of the γ-rays in that suitable exposure of sections of different thickness can be obtained at the same time. In the use of X-rays it is quite common to prepare the object so that one exposure will suffice. This may be done by filling up the low spots with a material having approximately the same absorption coefficient as the metal under investigation. In the case of aluminum, for example, the object may be immersed in carbon tetrachloride. If the authors

[2]Research laboratory, Aluminum Company of America, Cleveland.

could tell us what they think about the application of such methods in the case of γ-rays, I think that also would be of interest.

Authors' Closure (presented by R. F. Mehl)

It is a pleasure to reply to the kindly discussions presented by Messrs. Davey, Spring, Jesse, Alexander, Archer and Page. It is a rather difficult matter to present a work of this sort to the scientific public, for it proposes a new and thus far a relatively untried testing method somewhat similar in nature to an old and tried method, with distinctive characteristics a bit difficult to compare with the characteristics of the older method. It could be presented in a belligerent fashion, with every advantage on its side, or it could be tendered rather apologetically, with a hope that it might some day prove useful.

We have attempted to adopt a middle course—indeed, the only defensible one—in describing our work from a scientific viewpoint yet pointing out the practical features of the method which are even now clearly apparent. The adoption of this method in practice, or of any new testing method of proven efficiency, must depend entirely upon what business arrangements can be made by interested parties. Upon this latter point we can only lightly touch, leaving it to those who accomplish mysterious financial transformations far beyond the ken of the laboratory scientist.

It seems proper that Professor Davey should open the discussion. Although his comments require no answer it is pleasant to have his authoritative note.

Since the original work, reported in the present paper, was done, the Navy has had occasion to apply the method to one case of very considerable importance, and the results of this application have been so clear that we feel—in answer to Mr. Spring's discussion—that in one sense the method is no longer in an experimental stage. Whether or not the times of exposure are too long depends entirely upon the type of work to be done. For the mass inspection of such small articles as radio tubes or golf balls it may be that the method will not be especially useful, for the X-ray is so eminently successful in this field and the longer exposure times characteristic of the γ-ray method would make it seem cumbersome. But for the steel castings field particularly, the γ-ray method seems especially designed. In appraising the cost of the method and in studying the necessary exposure times, it must always be remembered that the complete portability of the necessary equipment saves both money and time, and that, furthermore, a large number of photographs can be taken at one time.

Although the authors are convinced that the exposure times now used are not prohibitively long for most work it would, naturally, be pleasant to decrease these times, as Mr. Spring and Mr. Jesse desire. We have made attempts in this direction with some promise of improvement, but have had little opportunity to make an extended investigation. The technique we have used is purely that devised by workers in the X-ray field during the last fifteen years. We have used X-ray films, X-ray developer, and X-ray

intensifying screens. It is quite possible that any or all of these three materials might be improved for γ-ray radiography.

The exposure times recorded in the present paper are purely incidental. We had but little upon which to approximate exposure times, and accordingly some exposure times were too short but most were far too long. Since this work was completed we have obtained some approximate data upon exposure times and it might be interesting to present at this time.

The figures given are all based upon 850 milligrams of radium or 850 millicuries of radium emanation. The distance from the source to the film in each case is the same, 18 inches.

Standard Time for Exposure
850 m.c., 18 inches source to film.

To show defects 2-4% of total thinckness:

Thickness	Enhancing Screens	Time of Exposure
1½"	2 films, 3 lead enhancing screens	12 min.
2⅜"	2 films, 3 lead enhancing screens	36 min.
4 "	2 films, 2 Patterson screens, and 1 lead screen	4 hours

To show defects 5-8% of total thickness:

Thickness	Enhancing Screens	Time of Exposure
1½"	2 films, 3 lead screens	7 min.
2⅜"	2 films, 3 lead screens	18 min.
4 "	2 films, 4 Patterson screens	1.3 hours

These figures are only approximate but we shall have accurately determined exposure curves to present later.[1]

The amount of radium emanation recovered daily from a radium salt in solution will vary, as Mr. Alexander points out, with the efficiency of the pumping apparatus. I am advised by Dr. West of the Howard Kelly Hospital that the simple apparatus used there—costing only about three hundred dollars—recovers from five grams of radium from 90 to 95 per cent of the total product of emanation. This would give 900 to 950 millicuries of radium emanation from one gram of radium instead of the 850 listed above.

It might be well at this time, leaving Mr. Alexander's discussion for a moment, to emphasize that all of the radiographic results we have obtained could have been obtained equally well from radium (the usual form in solution is radium bromide or in the solid radium sulphate). We have used radium emanation merely because it was more easily available to us than the radium itself.

In the practical application of this method doubtless occasionally the emanation will be the more convenient form and occasionally the radium salt. The two forms are equally good from the radiographic point of view.

The pumping of the emanation is a task requiring some care, as Mr. Alexander remarks. The operator at the Howard Kelly Hospital, however, recovers the daily crop of emanation from 5 grams of radium, concentrates it and seals it in glass capsules within 45 minutes, with practically no danger of loss. In twelve years this hospital has not lost one crop of emanation.

[1]Note added by authors November 23. The quantitative determination of the exposure conditions for γ-ray radiography is now completed and appears in the December, 1930, number of *Metals and Alloys*.

Although the pumping operation requires care, it is, therefore, by no means difficult or treacherous.

It is somewhat difficult to answer Mr. Archer's first question. The exposure curves for X-ray radiographs are best given upon the basis of milliampere-minutes exposure. The number of milliamperes sent through a 220,000-volt X-ray tube is usually five, so that the exposure for any thickness is read from the exposure curve (milliampere-minutes versus thickness of object) by dividing by five the number of milliampere-minutes corresponding to the thickness to be studied in order to get the number of minutes of exposure. Using the X-ray exposure curve given in the paper the exposure time for two inches of steel is about one minute, for three inches of steel about ten minutes, and for six inches of steel about six hundred hours.

The exposure for γ-rays is upon the basis of millicurie-minutes. The γ-ray method differs from the X-ray method in the fact that the intensity of the beam (millicuries for radium, milliamperes through the tube for X-rays) may be varied in their extreme limits merely by varying the amount of radium used. Upon the basis of the table just given the γ-ray exposure for two inches of steel is about 25,000 millicurie-minutes, for three inches of steel about 50,000 millicurie-minutes, and for six inches of steel about 600,000 millicurie-minutes. These exposures may be obtained either by using a weak source and a long exposure time, or a strong source and a shorter exposure time. If one gram of radium be used—or its equivalent in emanation—the time of exposure for two inches of steel becomes twenty-five minutes, for three inches of steel fifty minutes, and for six inches of steel six hundred minutes or ten hours.

Thus for six inches of steel the exposure time with one gram of radium or its equivalent of emanation is ten hours, whereas it is six hundred hours with the 220,000-volt X-ray tube. The relation is reversed with the thinner sections, for an unconscionable amount of radium would be required to get a radiograph through two inches of steel in the one minute required with the X-ray tube.

In making such a comparison, however, the practical features in the industrial application of the two methods must be kept in mind, especially the possibility of simultaneous photography with γ-rays and the relative unimportance of extremely short exposure times.

Mr. Archer's second question was partly answered by Mr. Alexander's discussion and by the reply to it. Briefly, the recovery operation is this: the radium is held as a solution of radium bromide in glass vessels; the spontaneously evolved gas—radium emanation or radon—is pumped by means of a Töpler pump first over potassium hydroxide to dry it and then into an eudiometer tube in which the admixed hydrogen and oxygen are removed by sparking; the partially purified gas is then collected in an inverted tube over mercury and transferred to another pumping apparatus in which the emanation is frozen from the admixed gases by liquid air; the admixed gases are then pumped away and the emanation allowed to vaporize through a thin capillary into a spherical glass bulb about an eighth of an inch in

diameter; this bulb is then sealed off and placed in a brass sheath to protect it, and the emanation is ready for use.

The danger in the use of radium emanation (or radium) is in two categories: first, that from the absorption by the body of the emitted radiations, alpha, beta, and gamma; and second, that from the absorption by the body of the radioactive matter itself. Most of the harm incurred by radium workers has come from the second, in which radioactive matter is taken into the body by careless handling. In radiography there is no danger of this sort, for the radium or the emanation is kept permanently sealed, and only the radiations escape.

To avoid harmful effects from these radiations the best precaution is absorptive screens and distance. The radioactive material should be kept in a lead box when not in use and the operator should not be near it except when necessary. Dangerous amount of exposure are well known by all hospitals using radium and can easily be avoided by the exercise of the obvious and simple precautions. Films carried in the pocket and developed from time to time will serve as an index of exposure. It can be confidently stated that the dangers from exposure in the practical use of radium for radiography, provided that the simple and known precaution be exercised, are very slight indeed. In fact, for radiography the handling of the radioactive material can be made much simpler than it possibly can in medicine, for the radioactive material may be kept safely in lead boxes until the objects to be inspected and the films are suitably arranged, then withdrawn with long-handled tongs and dropped into the holder provided, within a few seconds. With this technique the exposure of the operator is far too slight to be of any importance.

Mr. Archer's point in contrast and sensitivity is well taken. The diminished contrast we have found should, indeed, result in an inferior sensitivity. Despite this diminished contrast, however, we have been able to demonstrate the presence of defects 2 per cent of the total thickness, and it is probable that with machined surfaces a smaller defect might be found. We have stated that the sensitivity of the γ-ray method is about equal to that of the X-ray method only because the figure of 2 per cent is frequently given for X-ray radiography. It is quite possible that accurate determinations with the two methods with thickness differences less than 2 per cent should show an advantage for the X-ray method. But these minute differences are hardly of practical importance and we might therefore conclude that practically the two methods are of nearly equal sensitivity.

The use of such absorptive materials as carbon tetrachloride for aluminum and methyl bromide for heavier metals in order partially to eliminate the effect of irregular sections may be made in γ-ray radiography as well as in X-ray radiography. It is only when the piece to be inspected can be easily handled that this auxiliary technique may be applied, and unfortunately on large pieces it is ordinarily impossible to apply it, and in these cases the lack of sensitivity shown by the γ-ray to irregular sections—as illustrated in Figs. 9, 15, and 24—is of great advantage.

The behavior of absorptive materials in absorbing γ-rays is different

from that in absorbing X-rays, and an exactly parallel technique should not be used. This matter will be more completely discussed in a latter paper.

Mr. Page must have heard incorrectly, for we have never attempted to detect etching effects by γ-ray radiography. It is just possible that he confused the term echelon with etching. The development of higher voltage X-ray tubes will naturally make available a ray more penetrative than the present X-ray used for radiography. I am afraid that it will seem too much like defending the γ-ray method at any cost, yet it should be pointed out that higher voltage X-ray tubes will require much more expensive electrical equipment and will be more troublesome in operation, and also that the disadvantages in lack of portability and simplicity will still be present. To turn to Mr. Page's last comment, I am somewhat at a loss to understand the basis upon which he calculated the rather startling radium equivalent of a million volt X-ray tube, and I can only refer to the reply to Mr. Archer's discussion on this point.

It might be well in drawing this discussion to a close to again emphasize a few of the prominent features of the use of γ-rays in practical radiography, features which sharply distinguish it from the X-ray method. The γ-ray should not be directly compared with the X-ray method on the basis of exposure times, for the time of exposure is only a small fraction of the total time spent in the whole task of procuring a radiograph. Besides the actual exposure there is the task, when using the X-ray method, of bringing the casting to the X-ray equipment and afterwards of returning it to its proper place. These time-consuming tasks disappear in the γ-ray method for the necessary equipment is entirely portable. This fact, in addition, makes it possible to radiograph objects so inaccessible that the X-ray method could not possibly be used. The Navy has recently had occasion to study a large casting so placed that no method other than the γ-ray could have been used.

Furthermore, the possibility of taking a large number of simultaneous photographs alters the appraisal of time. We have applied the method practically in one case in which eight exposures on one casting were taken simultaneously. The total necessary exposure time for this task was one hour, with only about eight minutes equivalent time for one radiograph.

These various considerations illustrate the complications arising in comparing the two radiographic methods. For the practical application of the γ-ray method each industry must study its relation to the practical requirements to be met in that industry, and the decision between the X-ray method, and the γ-ray method must be drawn from a proper balancing of the practical advantages and disadvantages of the respective methods.

X-RAYS DETECT TWIN CRYSTALS IN ALPHA IRON

By N. P. Goss

Abstract

In a previous investigation it was found that when low carbon strip steel was drastically cold-rolled and heat treated at various temperatures a new orientation developed during the annealing operation.

The purpose of this paper is to show what conditions of cold rolling and heat treating are necessary in order to develop this new orientation.

IN a previous paper[1] it was found that when strip steel was given a reduction of 95 per cent and annealed at temperatures below the upper critical, the recrystallized grains were found to be oriented in such a way as to have the {211} lie in the direction of rolling. This annealing orientation was explained by assuming the ferrite grains to be twinned on the {211}.

When the grains were large a random grain structure resulted. This random distribution of the grains does not mean that they were not twinned, but the X-rays in such a case did not determine it.

Annealing above 1200 degrees Fahr. produced a structure composed of small grains, a great number of which were always found to be oriented with the {211} in the direction of rolling.

C. H. Mathewson and G. H. Edmunds[2] in their paper on "The Newman Bands in Ferrite" showed that the {211} were the twinning planes. Since the recrystallized grains in the strip steel used in this investigation tend to be oriented with the {211} in the direction of rolling, instead of the [110] it was possible to assume that this new orientation developed during recrystallization of the cold-rolled grains was due to the formation of annealing twins.

These annealing twins were never found unless the strip steel was cold-rolled rather drastically before annealing. In this strip steel

[1]Effect of Annealing on the Grain Structure of Extremely Hard-Rolled Steel Ribbon. N. P. Goss, TRANSACTIONS, American Society for Steel Treating, September, 1929, Vol. 16, p. 405.

[2]Technical Publication No. 139, American Institute of Mining and Metallurgical Engineers, Inc. October, 1928.

The author, Norman P. Goss, a member of the society, is associated with the Newburgh Steel Works, American Steel and Wire Co., Cleveland, Ohio. Manuscript received November 20, 1930.

annealing twins were only found when the area reduction was over 90 per cent.

In the case of strip steel only given an area reduction of 80 per cent and annealed, it was found that some of the grains retained the orientation produced by the cold rolling operation, but most of them were distributed at random. Fig. 1 is the X-ray pattern of a low carbon strip steel given an area reduction of 80 per cent and annealed at 1300 degrees Fahr. for 1 hour. Some of the grains have retained the orientation introduced by cold rolling, i. e., they are still oriented with the [110] in the rolling direction. The tendency of the recrystallized grains to retain this orientation is not marked in the case of flat wire. In round wires given an area reduction of 98 per cent it was found that a great many more grains retained this orientation [110].

It is interesting to note that this new orientation found in the low carbon rolled strip steel were never found in round wires made from the same steel drawn through wire drawing dies, especially in the range of area reductions necessary to produce this orientation. This means that the method of cold working used influences the kind of orientation to be expected in the annealed wire or strip.

In the case of strip steel given an area reduction of 92 per cent and annealed at various temperatures below the upper critical produced a grain structure having twin crystals, oriented in such a way as to have the {211} in the direction of rolling. Fig. 2 is the X-ray pattern of such a strip steel which was annealed at 1100 degrees Fahr. for 15 minutes. It shows that some of the grains have assumed this new orientation, but it can be seen that most of the grains are oriented at random.

Fig. 3 is the X-ray spectrogram of this same strip steel given an area reduction of 96 per cent and annealed at 1100 degrees Fahr. for 15 minutes. It shows that some of the grains have assumed this new orientation. By comparing Figs. 2 and 3 it will be seen that a greater number of grains are oriented so as to have the {211} in the direction of rolling.

When this strip steel was given an area reduction of 98 per cent and annealed at 1100 degrees Fahr. for 15 minutes an even greater number of grains possessed this new orientation. (See Fig. 4 and compare it with Figs. 2 and 3.) From this we see that the greater the reduction in area above 90 per cent the greater the number of twins which are oriented with the {211} in the direction of rolling.

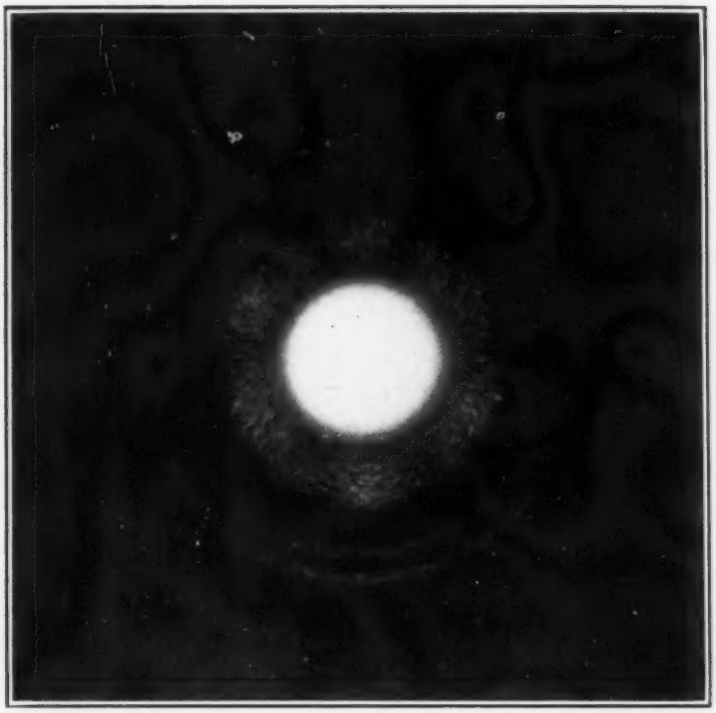


Fig. 1—Low Carbon Steel, Area Reduction 80 Per Cent, Annealed at 1300 Degrees Fahr. for 1 Hour.

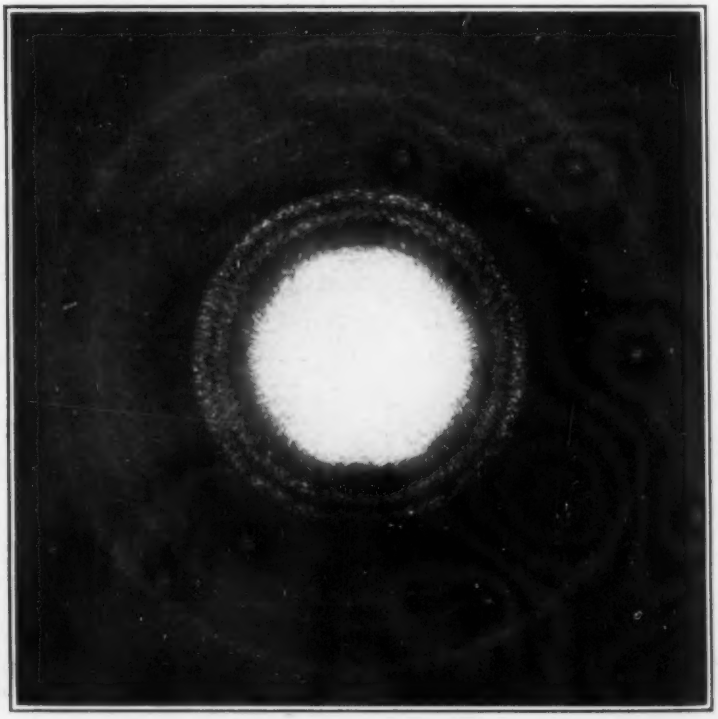


Fig. 2—Low Carbon Steel, Area Reduction 92 Per Cent, Annealed at 1100 Degrees Fahr. for 15 Minutes.

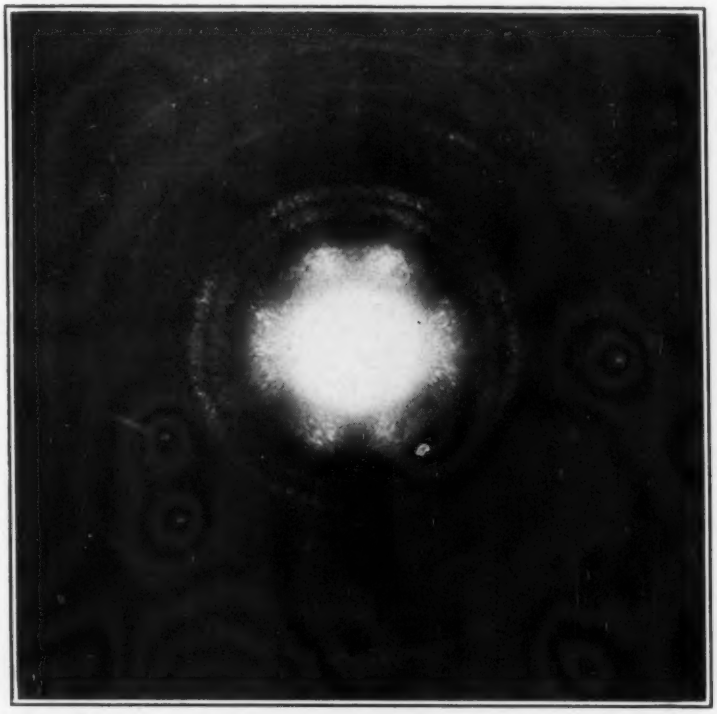


Fig. 3—Low Carbon Steel, Area Reduction 96¼ Per Cent, Annealed at 1100 Degrees Fahr. for 15 Minutes.

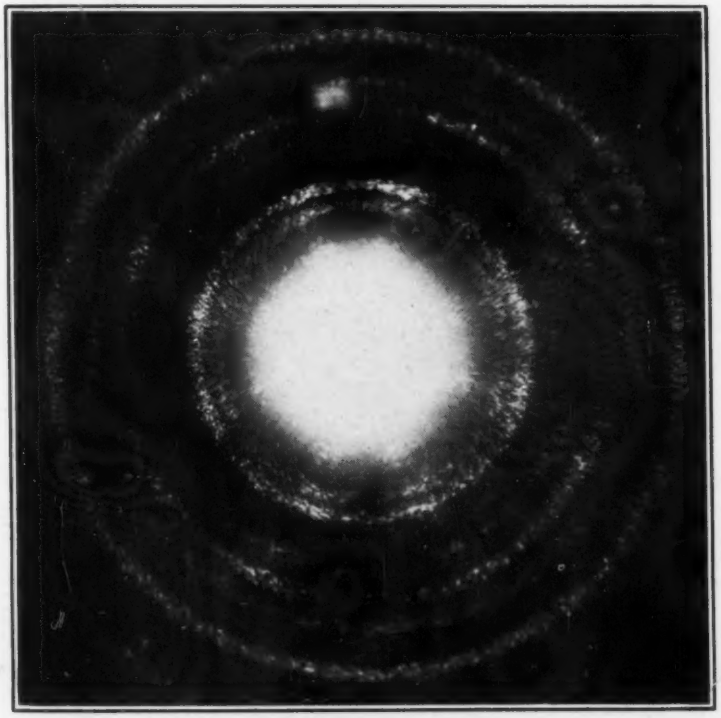


Fig. 4—Low Carbon Steel, Area Reduction 98 Per Cent, Annealed at 1100 Degrees Fahr. for 15 Minutes.

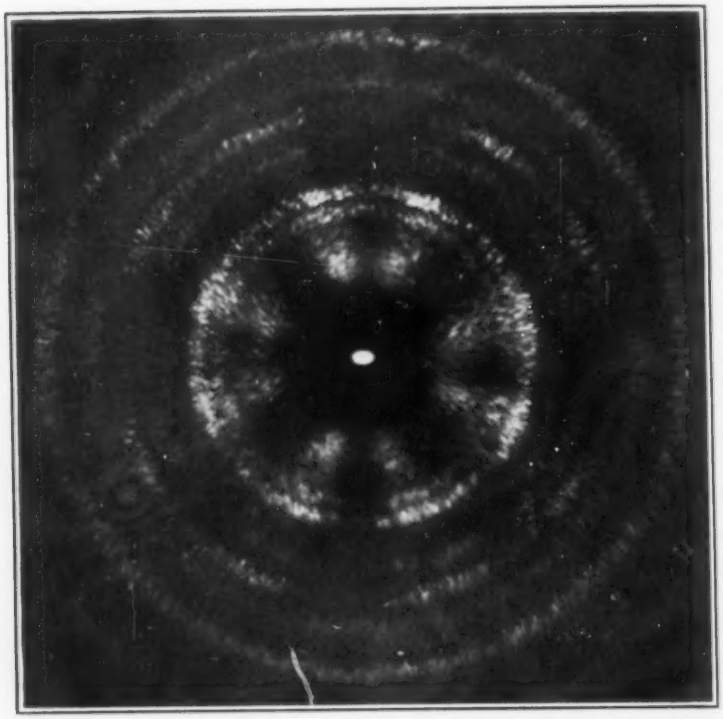


Fig. 4a—Low Carbon Steel. Area Reduction 98 Per Cent. Annealed at 1240 Degrees Fahr. for 15 Minutes.

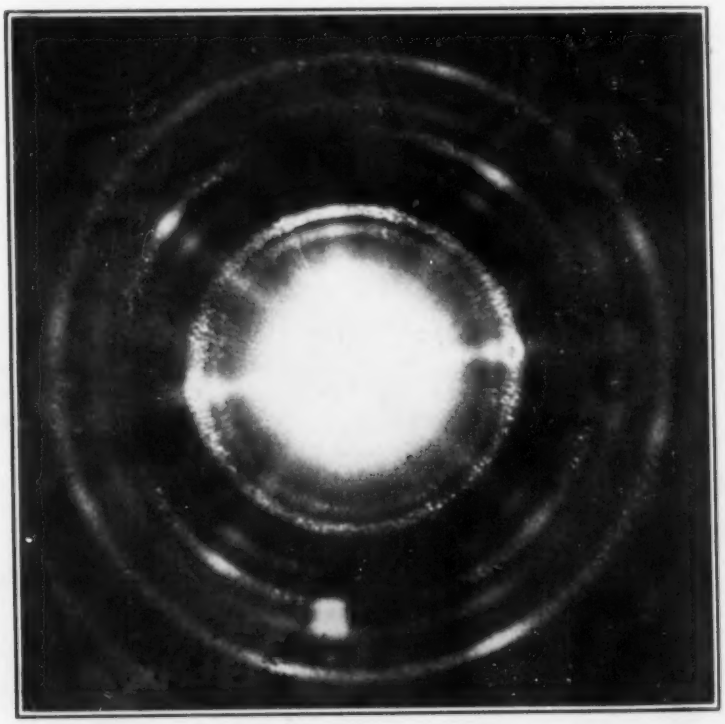


Fig. 5—Low Carbon Steel. Area Reduction 98 Per Cent. Annealed at 1000 Degrees Fahr. for 15 Minutes.

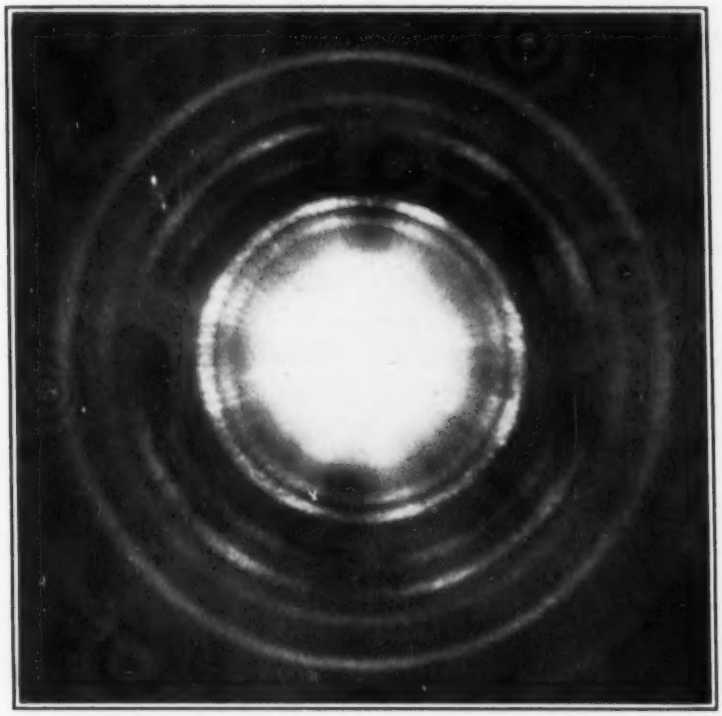


Fig. 6—Low Carbon Steel, Area Reduction 96¼ Per Cent. Annealed at 1250 Degrees Fahr. for 15 Minutes. Given Area Reduction 20 Per Cent After Above Heat Treatment.

Annealing at 1240 degrees Fahr. for 15 minutes seems to cause a greater number of these twins to be oriented in the direction of rolling. A comparison of Figs. 4 and 4a illustrates this. In Fig. 4 the diffraction rings tend to be continuous, in structure, i. e., the intensity maxima are not as sharply defined as they are in Fig. 4a. This would mean that the higher of the two temperatures is more favorable to the production of these annealing twins.

Fig. 5 is a very interesting X-ray pattern of this strip steel. In this case the steel was given an area reduction of 98 per cent and annealed at 1000 degrees Fahr. for 15 minutes. This time of annealing was not long enough to completely recrystallize the cold-rolled structure. It shows that the new grains are oriented so that the {211} lie in the direction of rolling, while the uncrystallized grains still remain oriented with the [110] in the direction of rolling.

It is also interesting to note that as long ago as 1910, Kroll[1] (*Journal,* Iron and Steel Institute 1919 I. pg. 304) observed that when a slightly strained cast silver was annealed, the grain structure was found to be twinned.

This means that twin bands are not formed during the cold working operation, but during recrystallization of the cold-worked structure. That Kroll's conclusions are correct, is proven in Fig. 5, which shows that the twins are produced during the annealing operation. This X-ray spectrogram shows that the recrystallized grains are twinned and that they tend to set the {211} in the direction of rolling.

Upon examining any of the X-ray patterns it will be seen that the diffraction arcs are not sharply defined, but are more or less broadened out.

If all the twins were oriented so as to have the {211} lie in the direction of rolling, then the diffraction spots due to the various planes (110), (100), (211), etc., would be short arcs instead of long arcs. This means that these twins are not oriented exactly as stated above, but deviate 10 degrees about this mean position, i. e., {211} planes may have at least this amount of variation from the ideal orientation.

Fig. 6 shows what takes place when a steel strip having its grains oriented so as to have the {211} in the rolling direction is given an area reduction of 20 per cent. This amount of area reduction did not change the orientation of the grains. Of course the grains are not completely fragmented, and it has been shown[3] that orientation of the grain fragments does not begin until they are completely fragmented.

Perhaps with greater area reductions, the grain fragments would be reoriented with the [110] in the direction of rolling. Unfortunately this area reduction was not even drastic enough to produce complete grain fragmentation. This steel strip could not be rolled down further than this because it was too thin to give it greater area reductions. It surely would be interesting to determine the point at which the {211} orientation changes over to the regular [110]. This transition, if it occurs, would take place only after the grains were completely fragmented. Grain size would therefore influence this transition point, which could be conveniently stated in terms of grain size and per cent area reduction.

Summary

Whenever low carbon steels were given area reductions greater than 90 per cent and annealed, a new orientation appeared. It was

[3]F. C. Elder, Iron and Steel Institute. N. P. Goss, "Applications of X-Rays to the Study of Plastic Deformation in Steel Wires and Molybdenum Ribbon." TRANSACTIONS, American Society for Steel Treating, February, 1930, Vol. 17, pp. 241 and 261.

explained if the ferrite grains are considered twinned on the {211}, and these planes tend to lie in the direction of rolling.

If the area reductions were less than 90 per cent no new orientation was found, after heat treatment, for area reductions between 80 and 90 per cent; it was found that after recrystallization some of the grains still retained the orientation developed by cold rolling, and that is the [110]. This tendency to retain the orientation developed during the cold rolling process is not marked in annealed steel strips.

The number of twin crystals oriented so that {211} lie in the direction of rolling increases with increasing reductions in area over 90 per cent, and also depends on the temperature at which these steel strips were annealed.

It was also found that these twin crystals are formed during the process of recrystallization, i. e., the grain which did not recrystallize retained the [110] in the direction of rolling.

This X-ray investigation is in agreement with numerous investigators in that twinning can occur in ferrite.

Cold rolling strip steels having this new recrystallization orientation with area reductions less than 20 per cent did not produce any change in the orientation. This means that these twins are quite stable when these steel strips are given moderate amounts of cold rolling. No doubt greater area reductions would have effected a reorientation.

THE PURIFICATION OF MANGANESE BY DISTILLATION

By James B. Friauf

Abstract

Metallic manganese of a high degree of purity has been obtained by the vacuum distillation of commercial manganese. The possible utility of vacuum distillation as a method for the purification of metals has long been recognized, and small amounts of some twenty-five metals were successfully purified in this way almost thirty years ago. The development of high speed vacuum pumps and of high frequency inductive heating has greatly facilitated the use of vacuum distillation for the preparation of very pure metals. Two furnaces have been constructed in this laboratory for the purification of manganese. Crude metallic manganese is melted by high frequency induction in an evacuated silica tube. Manganese vapor condenses on the relatively cool inner wall of a crucible inverted over the molten manganese. In a few hours a layer of distilled manganese several millimeters thick is formed and is removed when the furnace is cool. The distilled manganese contains about 0.004 per cent carbon, 0.002 per cent silicon, no iron, and only very slight traces of aluminum.

Introduction

SOME years ago the Bureau of Metallurgical Research undertook the preparation and study of iron-manganese and iron-manganese-carbon alloys of a high degree of purity. The metallic manganese that can be obtained commercially rarely contains more than 95 to 98 per cent manganese, the principal impurities being iron, carbon, silicon and aluminum. This material obviously is not suitable for the preparation of pure alloys and it was therefore necessary to find some way of preparing pure manganese from its chemical compounds or of purifying the crude metallic manganese that is available commercially.

A comprehensive study of a number of the methods that can

The author, Dr. James B. Friauf, is connected with the Bureau of Metallurgical Research, Carnegie Institute of Technology, Pittsburgh. Manuscript received May 26, 1930.

be used for the preparation of pure manganese has recently been made at the National Physical Laboratory in England.[1] Fairly promising results were obtained from the electrolysis of aqueous solutions of manganese salts, but experiments with the electrolytic method were discontinued when it was found that manganese of a high degree of purity could be produced by the vacuum distillation of the impure metal. Although distillation is usually thought of in connection with the purification of substances that are more volatile than the metals, it has long been used in the extraction of zinc and cadmium from their ores. The demand for high grade zinc during the war led to the use of a process in which crude zinc is purified by redistillation at atmospheric pressure.[2]

The advantages of vacuum distillation are two-fold. Distillation in a vacuum protects the metallic vapor and the condensed metal from oxidation or contamination by gases. In the second place, molten metals resemble other liquids in that they boil at a lower temperature under reduced pressure. Consequently distillation in a vacuum can be conducted at a lower temperature than would be required for distillation at atmospheric pressure. This is obviously an important consideration in the case of manganese which melts at 1244 degrees Cent. (2270 degrees Fahr.) and boils at 1900 degrees Cent. (3450 degrees Fahr.) under atmospheric pressure. These advantages have long been recognized and the possible utility of vacuum distillation for the purification of metals was pointed out almost fifty years ago.[3] By 1902, small amounts of some twenty-five metals including copper, gold, silver, iron and nickel as well as the more volatile metals like sodium, zinc, and cadmium, had been successfully purified in this way.[4] In recent years magnesium 99.99 per cent pure has been produced commercially by distillation or sublimation in a vacuum,[5] and zinc of a still higher degree of purity has been obtained by repeated distillation in a vacuum.[6] Al-

[1]Marie L. V. Gayler, *Journal*, Iron and Steel Institute, Vol. 115, 1927, p. 393.

[2]R. H. Engle, *Engineering and Mining Journal*, Vol. 102, 1916, p. 213. L. E. Wemple, *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 59, 1918, p. 171.

[3]A. Schuller, *Annalen der Physik*, Vol. 18, 1883, p. 317.

[4]Kahlbaum, Roth, and Siedler, *Zeitschrift für Anorganische Chemie*, Vol. 29, 1902, p. 177.

[5]H. E. Bakken, U. S. Patent No. 1,594,344, Aug. 3, 1926. H. E. Bakken and W. G. Harvey, U. S. Patent No. 1,594,345, Aug. 3, 1926. H. E. Bakken, *Chemical and Metallurgical Engineering*, Vol. 36, 1929, p. 345.

[6]H. M. Cyr, *Transactions*, American Electrochemical Society, Vol. 52, 1927, p. 349.

though manganese is appreciably less volatile than magnesium and zinc it has a vapor pressure of about 1.3 mm.[7] at its melting point. When the vapor pressure of a substance is known, the rate at which vapor leaves each square centimeter of the liquid surface can be computed from an equation given by Knudsen.[8] Using the vapor pressure of manganese at its melting point, it is found that approximately fifty grams of manganese are vaporized from each square centimeter of the liquid surface per hour. This figure indicates that manganese should distill quite rapidly at its melting point, and that there should be an appreciable amount of sublimation from solid manganese even before it is melted. The experimental results that have been obtained are in entire agreement with this conclusion.

High Frequency Furnaces for Vacuum Distillation

Although small amounts of many metals had been successfully purified by vacuum distillation over thirty years ago, the time required was so great as to seriously limit the practical usefulness of the method. Since then two technical developments have greatly simplified the experimental difficulties to be overcome in the vacuum distillation of metals. These are the invention of (1) high speed mercury vapor diffusion pumps which greatly facilitate the maintenance of high vacua, and (2) high frequency inductive heating which makes it possible to supply heat directly to the material that is to be heated. Both these developments have been used in the two furnaces constructed in this laboratory for the vacuum distillation of manganese.

Vertical cross sections through the two furnaces are shown drawn to the same scale in Fig. 1. The small furnace, which was constructed first, is not unlike the one that was used at the National Physical Laboratory,[1] but it has a number of refinements which make it possible to maintain a better vacuum than seems to have been obtained there. The furnace consists of a fused silica tube, A, four inches inside diameter and two feet long. Sand surface vitreosil tubing was used in the small furnace and molded silica pipe in the large furnace. Both proved to be satisfactorily vacuum tight. The lower end of the silica tube is sealed with vacuum wax to a water-

[7] International Critical Tables, Vol. III, 1928, p. 205.

[8] Knudsen, *Annalen der Physik*, Vol. 47, 1915, p. 697.

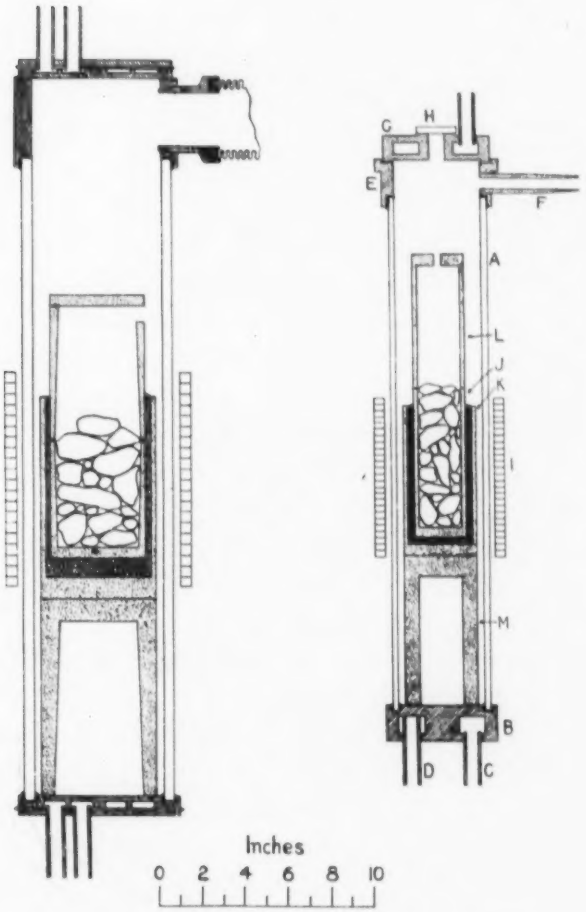


Fig. 1—Furnaces for the Vacuum Distillation of Manganese.

cooled end plate, B, with the inlet for the water at C and the outlet at D. The upper end of the tube is sealed to the brass ring, E, which is provided with a side tube, F, for connection to the vacuum pumps. Another water-cooled brass plate, G, which can be readily removed to permit access to the interior of the furnace, is seated on the upper surface of E. The ground joint between the two is made vacuum tight with stop-cock grease. A glass plate, H, covers a hole drilled through G and permits the interior of the furnace to be seen. I represents a water-cooled coil with thirty turns of flattened copper tubing. This is supplied with high frequency current from a 35 kilovolt-ampere converter.

Crude metallic manganese in lumps is placed in the pure magnesia crucible, J. This crucible is placed in a slightly larger crucible.

K, and the space between the two is loosely packed with 20 mesh alundum. Another crucible, L, which is similar to J except that it has a hole in the bottom, is inverted over J to condense the manganese vapor. The crucible, K, rests upon a magnesia support, M.

The furnace is evacuated by a mercury vapor diffusion pump backed by an oil pump and high frequency current from the converter is gradually supplied to the copper coil. The manganese in J is heated by induction; the rest of the furnace, being electrically non-conducting, is heated only by radiation or conduction from the hot metal. The manganese vapor from the molten manganese condenses on the relatively cool inner wall of the upper crucible, L, and forms a layer several millimeters thick. Heating is continued for a sufficient length of time to distill from about a third to a half the manganese charged in the lower crucible and the distilled metal is removed when the furnace is cold.

This furnace proved to be quite satisfactory except that its capacity was rather small. Also, on account of the small size of the inner crucible, J, the lumps of manganese frequently bridged across the upper end of this crucible and failed to melt down. For these reasons it was decided to build a larger furnace of the same general design and to secure a higher speed diffusion pump which would make it possible to maintain a better vacuum in the furnace.

The larger furnace, which is also shown in Fig. 1, has a silica tube with an inside diameter of six inches and a length of thirty inches. The brass ring at the upper end has been lengthened in order to introduce a larger connection to the vacuum pump. Relatively large amounts of gas are evolved when manganese is melted in a vacuum and to maintain a low pressure under these conditions it is essential to have a high speed vacuum pump and a large connecting tube between the furnace and the pump. Unless a large tube is used its resistance to the flow of gas at low pressures is sufficient to entirely neutralize the advantages of a high speed pump.[9] For this reason the inside diameter of the tube joining the furnace to the pump was increased to slightly over two and a half inches. Since a rigid connection between the furnace and pump is undesirable for mechanical reasons, a piece of flexible tubing was

[9]L. Dunoyer, *La Technique du Vide*, 1924, p. 133. G. W. C. Kaye, *High Vacua*, 1927, p. 169.

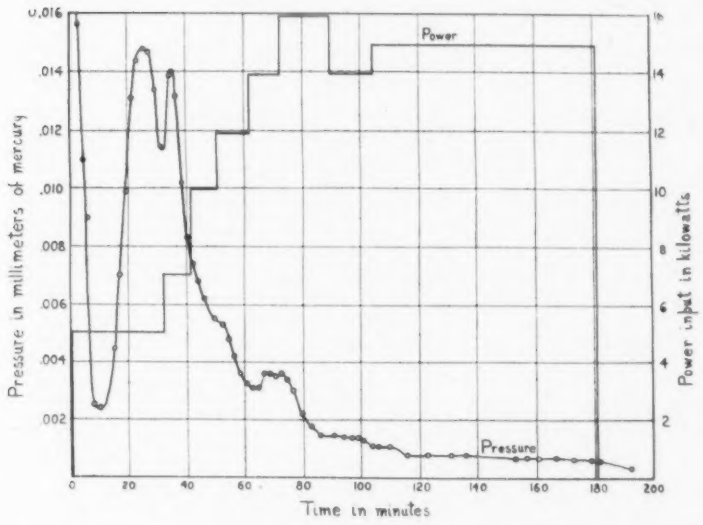


Fig. 2—Power Supplied to the Furnace and Pressure During a Vacuum Distillation.

used. The furnace is evacuated by a three-stage steel diffusion pump backed by a three-stage oil pump. A small hole in the brass ring at the upper end of the silica tube provides for a connection to the gage used to measure the pressure in the furnace. The pump and vacuum connections have proved highly satisfactory. Very little trouble has been experienced from leaks, and the high speed of the pump maintains a materially lower pressure than it was possible to obtain in the smaller furnace. The water-cooled coil for the larger furnace has thirty-eight turns of flattened copper tubing.

Fig. 2 is a graph showing the power supplied to the furnace and the pressure, plotted against the time. As power is supplied to the furnace its contents are heated, gas is driven off, and the pressure rises. If the power is kept constant the rate at which gas is evolved ultimately decreases and the pressure falls. The power is gradually increased to a value which experience has shown to be sufficient to melt the manganese and keep it molten. It will be noticed that the highest value reached by the pressure was only 0.015 millimeter and that at the end of the distillation it was less than 0.001 millimeter of mercury. In the distillation plotted in Fig. 2, 2788 grams of crude manganese were charged in the lower crucible, and 1010 grams of distilled metal were removed from the inner walls of the upper crucible.

Properties of Distilled Manganese

A spectroscopic analysis of the distilled manganese showed no iron and only very slight traces of aluminum. Chemical analyses gave 0.004 per cent carbon and 0.002 per cent silicon. These results are in complete accord with those of the other investigators who have found that a single vacuum distillation produces metal of a high degree of purity.

Distilled manganese has a beautiful silvery gray luster which is quite resistant to atmospheric corrosion. Samples exposed to the laboratory air for over a year are only slightly tarnished. The distilled metal is very brittle and hard enough to scratch glass. It combines readily with nitrogen when heated; consequently pure manganese alloys cannot be prepared by melting in a nitrogen atmosphere. Vacuum melting of iron-manganese is also impracticable because of the excessive loss of manganese by volatilization. The alloys which have been prepared with distilled manganese have been melted in argon.

It is a pleasure for the writer to acknowledge his indebtedness to Dr. F. M. Walters, Jr., for his assistance during this work, and for the spectroscopic analysis of distilled manganese, and to Maxwell Gensamer for the chemical analyses for carbon and silicon.

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